A HANDBOOK

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A HANDBOOK

FOR

CEMENT WORKS CHEMISTS

BY

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AND CONSULTING CHEMIST.

SECOND EDITION, REVISED.

WITH ILLUSTRATIONS.

OF INDUSTRIES
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1917.

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PREFACE TO SECOND EDITION.

THE publication of a Second Edition of this Handbook has been withheld for some considerable time owing to the war, but constant inquiries for it indicate that it is valued and would prove of service. The work has been thoroughly revised, much of the descriptive text entirely re-written, and many Sections necessarily extended by the inclusion of recent practice. Additional chapters are given upon "The Analysis and Examination of Aggregates, &c.," and "Notes on Cement Testing," whilst the Tables of Atomic Weights and Factors for use in Analysis have been revised in accordance with the International Table of Atomic Weights, 1916, thus bringing the book up to date. The Author's thanks are expressed to Mr. J. T. Stiff, for help in the revision, also to Mr. J. Crabb (Swanscombe), and Mr. W. F. Gibson, as well as to those authors previously acknowledged.

F. B. G.

HORNCHURCH, ESSEX,
December, 1916.

PREFACE TO FIRST EDITION.

THE Author wishes to acknowledge his indebtedness to Messrs. H. W. Anderson, C.E., H. K. G. Bamber, F.C.S., J. W. Gatehouse, F.I.C., H. H. Slater, F.I.C., and J. T. Stiff, the authors of the volumes consulted, and others who have materially assisted him during the compilation of this handbook.

KINGSTON-ON-THAMES,
1908.

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CEMENT CHEMISTS' HANDBOOK.

CHAPTER I.

INTRODUCTION.

As indicated by its title, this book is intended primarily for Cement Works Chemists, although it is hoped that it may be found of use in other laboratories. It has been the aim of the compiler to include only essential details of manipulation where this was possible without ambiguity. Each separate process will be found in a numbered paragraph, and a method once described is indicated in the text by that number when it has again to be carried out. An epitome of most of the processes will be found at the foot of the fuller description. In this way the book may be of use as a guide to younger and less experienced chemists, as well as for reference purposes by the experienced.

For the sake of uniformity the strengths and volumes of the solutions employed are stated according to Reddrop's system, as described and followed by Phillips in his *Engineering Chemistry.*

A table will be found in the Appendix giving instructions for making all the necessary reagents and their strength in terms of E, this being the symbol used [illegible]e reagent by the equivalent or equivalents of th[illegible] in grams per litre. These reagents need not be [illegible] with the same degree of accuracy as those employed in volumetric analysis. For fuller particulars reference should be made to the work mentioned.

The following is a list of chemicals and apparatus required for the processes described in this handbook:—

CHEMICALS REQUIRED.

Pure hydrochloric acid.
Commercial „ „
Pure nitric acid.
„ sulphuric acid.
„ acetic acid.
„ hydrofluoric acid.
Oleic acid.
Oxalic acid.
Pyrogallol.
Pure ammonium hydrate (·880).
„ „ oxalate.
„ „ thiocyanate.
Ammonium carbonate.
„ chloride.
„ molybdate.
Sodium carbonate (anhydrous).
„ „ (crystals).
„ bicarbonate.
„ hydrogen phosphate.
„ thiosulphate.
„ hydrate.
Sodium acetate.
„ sulphite.
Soda lime.
Potassium bichromate.
„ chromate.
„ ferricyanide.
„ ferrocyanide.
„ nitrate.
„ permanganate.
„ carbonate.
„ hydrate.
„ bisulphate
„ chlorate.
Ferrous ammonium sulphate.
Magnesium chloride.
„ oxide.
Manganese carbonate.
Copper sulphate.
„ oxide.
„ turnings.
Silver nitrate.

CHEMICALS REQUIRED—(*continued*).

Ferrous sulphide.
Piano wire.
Indigo.
Mercuric chloride.
Bromine
Distilled water.
Calcium chloride.
,, carbonate.
Iceland spar.
Barium chloride.
Platinum chloride.
Lead nitrate.
Pure zinc.
Litmus.
Methyl orange.
Phenolphthalein.
Alcohol.
Ether.
Paraffin.
Petroleum ether.

APPARATUS REQUIRED.

Abel's flash point apparatus.
Absorption apparatus.
Accurate balance and weights.
Agate pestle and mortar.
Air oven.
Argand burners.
Barometer.
Battery.
Beakers.
Bellows.
Blowpipe.
Bottles.
Bunsen burners.
Burette holders.
Burettes.
Calcimeters.
Calorimeter.
Clips.
Clock glasses.
Condensers.
Cork borer.
Corks.
Crucibles.
Desiccator.
Drying tubes.
Erlenmeyer flasks.
Evaporating basins.
File.
Filter paper.
Flasks.
Funnels.
Gas analysis apparatus.
Retort stands.
Glass tubing and rod.
Graduated flasks.
Grease pencil.
Hot plate.
India-rubber tubing.
Iron pestle and mortar.
Measuring tubes.

APPARATUS REQUIRED—(*continued*).

Microscope.
Muffle furnace.
Nitrometer.
Phillips beakers.
Pipe-clay triangles.
Pipettes.
Platinum crucible and capsule.
Reagent bottles.
Retorts.
Rough balance.
Rubber bungs.
Sample jars.
Sieves.
Specific gravity bottles.
Spatulas.
Spotting tile.
Thermometers.
Test tubes.
Viscosimeter.
Ware pestle and mortar.
Wash bottles.
Watch glasses.
Weighing bottles.

The following works have been consulted in the compilation of this book, and are recommended for reference purposes :—

AUTHORS.	BOOK.
Phillips.	*Engineering Chemistry.*
Stanger and Blount.	Reprint from *J.S.C.I.* on Cement Analysis.
Clowes and Coleman.	*Quantitative Analysis.*
C. and J. J. Beringer.	*A Text Book on Assaying.*
Dennis.	Hempel's *Gas Analysis.*
Kenwood.	*Public Health Laboratory Work.*
Bayley.	*Chemist's Pocket Book.*
Meade. (U.S.A.)	*Portland Cement.*
Eckel. ,,	*Limes, Plasters, and Cements.*
Taylor. ,,	*Cement Testing.*
Orton. ,,	*Hydraulic Cements* (Ohio Survey, vol. iii.).
Mellor, J. W.	*A Treatise on Quantitative Inorganic Analysis* (Griffin).

CHAPTER II.

ANALYSIS OF RAW MATERIALS.

Sampling.—When sampling a new supply, or possible source, of raw materials, great care must be taken to obtain an accurate average sample.

When possible the sampling should be supervised by the chemist or other competent person. The plan to be followed necessarily varies with the situation and material, and must be left to the discretion of those on the spot. If possible, samples should be taken at different depths as well as in different places on the surface of a deposit. Each sample should be carefully numbered and labelled for reference purposes. Distinct geological layers should be specially sampled and separately analysed.

All the samples should be examined separately, and an average sample of the lot should also be made and analysed. In the case of large samples of fairly dry materials, such as limestone, the material should be reduced to a convenient size, thoroughly mixed, made into a heap, and sampled by "quartering"; that is, the heap is divided into four equal parts diagonally, and an equal amount taken from each part; if necessary, this may be repeated until a convenient weight or bulk is obtained. In the case of wet clays, etc., greater difficulty will be experienced, and these are more readily sampled after being allowed to partially dry. If the percentage of moisture in sample is required, however, this should be done approximately using as large a sample as possible, as well as more accurately, as described later.

(1) **Chalk or Limestone.**—If sufficiently dry, break up the sample in a clean iron mortar, and, by quartering, obtain a portion for examination weighing about 300 grams. Store the remainder in a numbered and labelled jar or large bottle for reference purposes. Grind the smaller portion by hand or in a clean sample mill to all pass through the 100-mesh sieve, and place in a clean, dry, stoppered bottle for analysis (A).

(2) **Moisture.**—If very wet and difficult to reduce to powder, weigh up on a "decimal" or other balance as large a quantity as possible, say 1,000 grams, of the roughly crushed sample. Place in a large tin dish or tray and dry over the hot plate, taking care that the temperature does not rise above 110° to 120° C. When apparently quite dry allow to cool, and weigh.

Loss in weight = approximate moisture.

Repeat if necessary, calculate to percentage.

As a rule a very approximate estimation of the moisture only is necessary. Treat the dry sample as in (1).

(3) If fairly dry and readily powdered, an accurate estimation of moisture may be made as follows :—

Weigh out accurately into a flat porcelain weighed dish or capsule 5 grams of the sample (A) and place in a steam or hot air oven at a temperature of 100° C. for one hour. Remove from oven and place in a desiccator; when quite cool weigh rapidly; note weight. Generally one hour's drying is sufficient, but the result should be checked by replacing in the oven for another twenty minutes, cooling and weighing as before.

Loss in weight × 20 = percentage of moisture at 100° C.

Place the dried material in a clean, dry, stoppered weighing tube, and use for analysis.

Epitome.

Break up sample roughly.
" Quarter " to about 300 grams.
" Approximate moisture " on hot plate.
Accurate moisture in oven at 100° C.
Reserve large sample for reference, dried material for analysis.

(4) **Loss on Ignition**.—Into a weighed platinum capsule or crucible weigh out 0·5 gram of the dry material and place in a gas muffle at a

bright red heat for twenty minutes. It is sometimes necessary to cover the capsule with a crucible lid during the first five minutes of heating to avoid loss by "spurting." At the end of the twenty minutes remove from muffle and allow to cool in a desiccator; weigh.

Loss in weight × 200 = loss on ignition (or $CO_2 + H_2O$ and organic matter).

Reserve residue for analysis.

Epitome.

0·5 gram in muffle for twenty minutes.
Cool in desiccator and weigh.

(5) **Silica and Insoluble.**—Brush the ignited residue from (4) into a 6- or 7-inch evaporating dish, flat form. Cautiously add about 15 c.c. of distilled water, rotate to prevent formation of lumps, and add 25 c.c. of 10E HCl; wash out the platinum vessel with a little acid into the dish, and place latter on the cooler part of a hot plate or on a water bath.

Evaporate very carefully to dryness to avoid spurting, and then cover with a clock glass and remove to the hottest part of plate and allow to bake for one hour. Remove from hot plate, allow to cool for five minutes and add 25 c.c. of 10E HCl and warm very gently until the residue is free from colour due to iron compounds. Wash down clock glass and interior of dish with boiling water from a wash bottle. Filter through a 9- or 12-cm. "rapid" paper into a 40-oz. Phillips beaker, retaining as much as possible of the residue in the dish; wash by decantation thus three times at least, and then wash residue into the filter, scrub out the dish by means of a rubber-tipped glass rod, and finally with the tip of the finger, until every trace of residue is contained in the filter paper.

When filtrate has run through, place the funnel and contents, which, as well as all other apparatus used, should be distinguished by a number or mark, in a drying cone on the hot plate or oven to dry. Carefully transfer paper and contents to a weighed and marked porcelain crucible, burn off paper over a burner or in the mouth of the muffle furnace, then ignite in muffle for one hour. Cool in desiccator and weigh.

It is convenient to retain all ppts. obtained during analysis for ignition together later.

Weight − weight of crucible = insoluble.

Insoluble × 200 = per cent. silica + insoluble.

Epitome.

To residue from (5) add 25 c.c. 10E HCl, evaporate to dryness, and bake.

Take up with 25 c.c. HCl. Filter, wash, weigh. Reserve filtrate for (7).

(6) It is not generally necessary to separately estimate the soluble and insoluble SiO_2, but this

may be done on another portion of the original material as follows:—Weigh out 0·5 gram into a 5-inch evaporating dish, add water as before, and cautiously add 25 c.c. 10E HCl, covering the dish as much as possible with a clock glass to prevent loss. Warm on a hot plate until all soluble matter is in solution, then filter this through 9-cm. rapid paper and wash by decantation, retaining the residue in the dish. The filtrate is used for SO_3 estimation (13).

Invert the funnel over the dish and wash out any trace of residue in the paper. Add 10 c.c. 3E Na_2CO_3 solution and boil for ten to fifteen minutes, filter rapidly through the paper previously used and wash with boiling water until free from alkali or until a drop of filtrate evaporated on a watch glass leaves no residue. Dry, ignite, and weigh.

Weight × 200 = per cent. insoluble residue.

It may be necessary to estimate alumina and iron oxide in the insoluble residue by "fusion" or after treatment with HF.

Epitome.

0·5 gram treated with 25 c.c. HCl warm.
Filter by decantation, wash.
Boil residue with 10 c.c. Na_2CO_3 solution.
Filter, wash, dry, weigh.

(7) **Alumina and Iron Oxide.**—The filtrate from (5) is returned to the dish and warmed until

nearly boiling. Add a drop or two of bromine water; continue to warm, and add carefully 10E NH_4OH until a ppt. forms; stir with glass rod and add slight excess. Allow to remain on hot plate until nearly free from odour of ammonia, or about fifteen minutes. Filter through a 15-cm. rapid paper and wash with boiling water, churning up the ppt. on the filter paper with the water jet, until a drop of the filtrate gives no indication of the presence of chloride when tested with $\frac{E}{5}$ $AgNO_3$ solution. Dry and ignite in muffle one hour, cool in desiccator, and weigh.

Weight of ppt. × by 200 = per cent. of $Al_2O_3 + Fe_2O_3$. The amount of $Al_2O_3 + Fe_2O_3$ in a limestone is generally so small as to render separation unnecessary.

Epitome.

Filtrate from (5); nearly boil.
Oxidise with Br water and ppt. with NH_4OH. Warm for fifteen minutes, filter, wash, ignite, weigh.

(8) **Lime.**—The filtrate from (7) is brought nearly to boiling point in a large Phillips beaker. Add a few c.c. of 5E NH_4OH and boil; whilst boiling, add 50 c.c. of boiling $\frac{3E}{5}$ Am. oxalate solution, place a watch glass over beaker and allow to steadily boil for five minutes. Remove from the

direct heat and allow to settle, if possible in a warm place, for one hour or longer.

Filter through a close-textured 15-cm. paper into a large bottle or beaker, wash the ppt., first by decantation with warm but not boiling water; then wash ppt. into the filter paper, removing any traces adhering to sides of the beaker by means of a rubber-tipped glass rod.

The lime may be determined gravimetrically as CaO (8*a*), $CaCO_3$ (8*b*), or $CaSO_4$ (9), or volumetrically by means of standard permanganate (10).

(8*a*) Gravimetric Determination as CaO.

—Wash the ppt. until free from chlorides, dry, ignite in platinum crucible, at first over bunsen then over a blast burner, or in hot muffle furnace, cool in desiccator and weigh, repeat ignition until a constant weight is obtained.

Weight of $CaO \times 200$ = per cent. CaO.
Per cent. $CaO \times 1{\cdot}786$ = per cent. $CaCO_3$.

(8*b*) Gravimetric Determination as $CaCO_3$.

—Wash ppt. until free from chlorides, dry, and ignite, preferably in a platinum crucible, over a bunsen burner, allow to cool, and moisten contents with 5E Am_2CO_3, carefully drive off moisture, and again ignite over bunsen. Cool in desiccator and weigh as $CaCO_3$.

Weight of $CaCO_3 \times 200$ = per cent. $CaCO_3$.
Per cent. $CaCO_3 \times 0{\cdot}56$ = per cent. CaO.

(9) **Lime as $CaSO_4$.**—Wash calcium oxalate ppt. until free from chlorides, dry, place in weighed platinum crucible with a lid, and ignite gently over a bunsen burner to burn off filter paper.

Remove from burner, allow to cool, and add by means of a pipette or glass tube with a fine point a few drops of water, then, drop by drop, pure strong (36E) H_2SO_4, holding the lid over the crucible whilst so doing, until the whole of the ppt. is moistened with the acid. Heat the uncovered crucible over an argand or bunsen burner with a rose top, using a very small flame, in a draught cupboard until fumes cease to be evolved. Replace the lid and heat just to redness over a burner for about thirty seconds, cool and weigh as $CaSO_4$.

Weight of $CaSO_4 \times 0{\cdot}412 \times 200 =$ per cent. CaO; or use table (see *Appendix*). Calculate to $CaCO_3$ as in (10).

(10) **Volumetric Determination of CaO.**—In order to avoid accumulation of a bulky filtrate for MgO determination, continue washing the calcium oxalate ppt. on the paper into another vessel until a drop of the filtrate will not discolour a little distilled water rendered just pink with a trace of permanganate solution acidified with 5E H_2SO_4.

Then carefully remove the paper and its contents from the funnel, open it out over a large ordinary beaker, and wash the ppt. from the paper into the beaker; it is better to complete this operation by using a small wash bottle containing 5E H_2SO_4. Add 15 to 20 c.c. of this acid, make up the contents of beaker with water to about 250 c.c., and bring to boiling point.

Titrate with standard permanganate solution, the strength of which is known in terms of CaO, until a permanent pink colouration is obtained.

Number of c.c. used × factor × 200 = percentage of CaO.

Per cent. CaO × 1·786 = $CaCO_3$.

Epitome.

(8) Boil filtrate (7); add ammonia + 50 c.c. boiling Am. ox.; allow to settle, filter, wash, ignite, and dry as CaO (8*a*) or $CaCO_3$ (8*b*).

(9) Dry, ignite, add sulphuric acid in platinum crucible, drive off sulphuric acid, weigh as $CaSO_4$.

(10) Dissolve in 5E H_2SO_4 + H_2O, boil, titrate with standard potassium permanganate.

(11) **Magnesia.**—Evaporate the filtrate from (8) nearly to dryness in the large dish; add 30 c.c. 16E HNO_3 and heat on hot plate in draught cupboard until no more ammoniacal salts are volatilised.

Remove from plate, allow to cool; then add about 5 c.c. of 10E HCl and about 20 c.c. of water. Warm and add slight excess of 10E

NH_4OH; filter off the small ppt. of SiO_2, Al_2O_3, Fe_2O_3 through a 5 cm. rapid paper and wash. This may generally be ignored as being due to impurities in the reagents and from the vessels used.

To the filtrate, which should not exceed 100 c.c. or so, when cool add at least 10 c.c. of 20E NH_4OH, then 5 c.c. of $\frac{2E}{3}$ Na_2HPO_4 solution. Stir well with a rubber-tipped rod, but avoid touching the sides of the beaker as much as possible, and place in a dish of water or other cool place to settle for twelve hours (over night) if time permits.

When the ppt. has completely settled, filter through a 7- or 9-cm. close-texture filter paper, wash by decantation, using 5E NH_4OH; then transfer ppt. to the paper, rub out carefully any particles adhering to side of beaker, and wash on the paper until a drop of the filtrate, when acidified with HNO_3, gives no ppt. with $\frac{E}{5}$ $AgNO_3$ solution.

It is important that the washing should be complete, but not excessive.

Ignite, best in a platinum crucible, first at a low temperature over a bunsen burner and then at a higher temperature; or in a muffle furnace for twenty minutes. Cool in a desiccator and weigh as $Mg_2P_2O_7$.

Weight of $Mg_2P_2O_7 \times 0{\cdot}362 \times 200 =$ per cent. MgO; or use table (*Appendix*).

Per cent. $MgO \times 2{\cdot}1$ = per cent. $MgCO_3$.

Sodium ammonium phosphate (microcosmic salt) has been recommended in place of sodium hydrogen phosphate.

Epitome.

Evaporate lime filtrate to dryness.
Add 30 c.c. 16E HNO_3 and drive off ammonium compounds.
Take up with 5 c.c. 10E HCl, ppt. with 10E NH_4OH.
Filter, wash, cool. Add 20 c.c. 10E NH_4OH and 5 c.c. Na_2HPO_4 solution, stir.
Allow to settle, filter, wash with 5E NH_4OH.
Dry, ignite, weigh as $Mg_2P_2O_7$.
Calculate to MgO or $MgCO_3$.

(12) Alkalies are generally estimated by difference, but the method described under Clay (31) or Cement (122) may be adopted if necessary.

(13) Sulphates.—Boil the filtrate from (6) and, whilst still boiling, add 10 c.c. of E $BaCl_2$ solution; after five minutes allow to settle in a warm place for a few hours. Filter through a 7-cm. "sulphate" paper, wash with warm water until free from chloride, dry, ignite, and weigh as $BaSO_4$.

Weight of $BaSO_4 \times 0{\cdot}3431 \times 200$ = per cent. of SO_3 (see *Appendix* 26A).

Epitome.

Filtrate from (6). Boil, add 10 c.c. E $BaCl_2$. Allow to settle, filter, wash, ignite, weigh as $BaSO_4$.

(14) **Accurate Estimation of Carbon Dioxide.**—One gram of the limestone is decomposed by hydrochloric acid in a flask, the carbon dioxide evolved is absorbed by potash solution, after passing over various reagents, and weighed.

The following apparatus must be fitted up. The lettering refers to the illustration. (Fig. 1.)

A fairly wide-mouthed flask (*A*) is fitted with a three-holed rubber bung; through one hole is passed a stoppered funnel (*b*); a tube (*a*), containing soda-lime, is passed through one of the other holes; and the remaining one is fitted with a glass tube which is connected to the absorption apparatus. A two-holed bung may be used, in which case the funnel is substituted by a pipette connected at the upper end with a soda-lime tube, a clip is placed on the connecting rubber, and this is released when the acid has to enter the flask. The tube leading from the flask is connected by the rubber tubing, first with bulbs (*B*) containing strong H_2SO_4; secondly, to a U-tube (*C*), the nearer limb of which is filled with copper sulphate pumice, and the other with solid calcium chloride. Connected to this tube are the potash bulbs (*D*), in which the carbon dioxide has to be absorbed and weighed; and these are in turn connected to a straight tube containing calcium chloride, which is weighed with the bulbs.

The absorption bulbs should be filled with

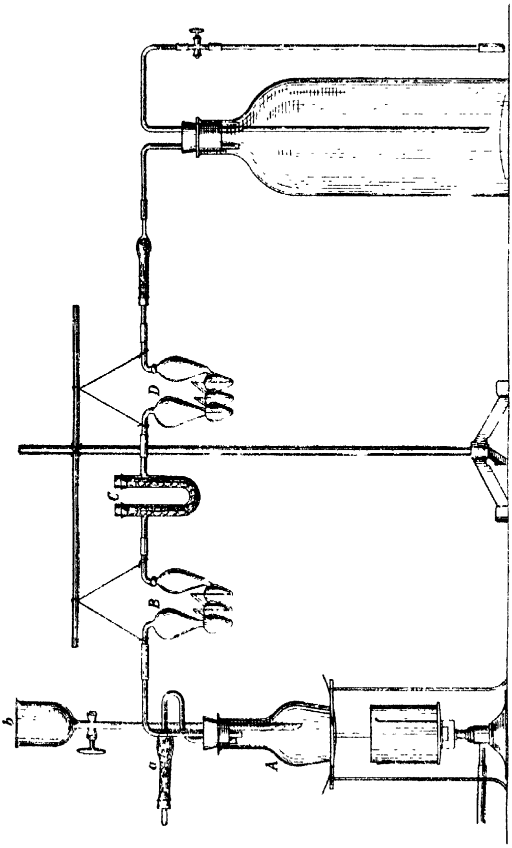

Fig. 1.—Apparatus for Estimation of CO_2.

5E KOH solution in sufficient quantity to half fill the smaller bulbs and to be contained in the large pear-shaped bulb should there be any back pressure. The whole apparatus is connected with an aspirator made from a Winchester quart bottle as shown.

Instead of potash bulbs, a U-tube containing soda-lime may be used.

Stoppered U-tubes are to be preferred to corked ones ; if the latter are used, they should be made air-tight with paraffin wax.

A little cotton-wool should be placed before and after the calcium chloride and pumice-stone in the tubes, to prevent the passage of fine dust.

All rubber connections should be wired on and the apparatus tested before use. When the apparatus is ready for use, detach the absorption apparatus ; close the open end of the rubber tube by means of a bit of glass rod. See that the bulbs and tube are quite dry, and weigh.

Weigh into the flask 1 gram of the material, cover it with water. Place 50 c.c. of 5E HCl in the funnel or pipette, and reconnect up the whole apparatus, except the aspirator. Close the air inlet through the soda-lime tube, by means of rubber tubing and clip.

Allow acid to drop slowly on the carbonate so that the bubbles of air driven from the apparatus may be easily counted as they pass through the sulphuric acid ; continue addition of the acid until effervescence ceases.

Close the stopper of the funnel, or, if a pipette, carefully push the point under the surface of the liquid, attach the aspirator and set it in action. Place an argand burner under the flask and warm gently. Open the air inlet tube so that a current of air is made to pass through the apparatus until quite half the water in the aspirator has run out. Then stop the operation, disconnect and stopper up the weighed bulbs, allow to get quite cool, and weigh.

Increase in weight × 100 = per cent. CO_2.

Epitome.

One gram decomposed by hydrochloric acid in the absorption apparatus.

Carbon dioxide absorbed in potash or soda-lime, and weighed.

(15) **Rapid Estimation of Carbon Dioxide.**—A rapid and fairly accurate estimation of carbon dioxide may be made in the following apparatus, represented in Fig. 2.

Fit a wide-mouthed 4-oz. flask with a rubber bung. Through one hole place a piece of glass tube attached to a straight-drying tube. Through the other hole run a piece of glass tubing closed at the outer end with a small piece of rubber tubing and glass rod.

A small piece of cotton-wool is first placed in the bulb tube, and the bulb is then filled with dehydrated copper sulphate pumice followed by granular calcium chloride in the straight part; another piece of wool is then inserted and the tube closed with a cork bearing a small glass tube.

Thoroughly dry the apparatus, weigh out 1 gram of the carbonate and brush it into the flask ; cover with water, and then lower a small glass or gutta-percha test tube containing about 7 c.c. of 5E HCl into the flask by means of a piece of cotton. Insert the stopper so that the cotton from which the test tube is suspended is held in place.

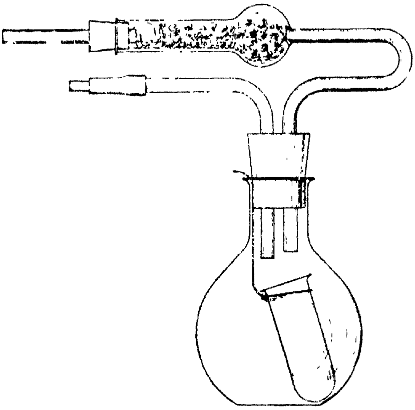

Fig. 2.—Apparatus for Rapid Estimation of CO_2.

Weigh the whole apparatus and contents. Slightly tip the apparatus so that the acid is caused to leave the test tube a little at a time. When effervescence has ceased, warm the flask over an argand burner for about five minutes ; allow it to cool somewhat, and then draw, by means of an aspirator as in (14) or by the mouth, a current of air through the apparatus, for this purpose removing the rubber cap of the inlet tube. Allow the apparatus to cool, see that the exterior is quite dry, and weigh complete as before.

Loss in weight × 100 = per cent. CO_2.

A determination can be made in about thirty minutes.

A modified Schrötter's apparatus may be employed in place of above if preferred.

Epitome.

Weigh into prepared flask 1 gram.

Decompose with hydrochloric acid, warm, cool, weigh.

The total carbonate in an ordinary chalk or limestone may be rapidly estimated as calcium carbonate upon one of the calcimeters described in Chapter III., and especially conveniently upon Slater's instrument, using the table given in *Appendix* (27A and 28A).

A dolomitic or magnesian limestone is insoluble in cold hydrochloric acid, and therefore the carbon dioxide cannot be estimated upon a calcimeter. Of course, for Portland cement manufacture to standard specifications such limestones are useless, and therefore of no importance.

Should an analysis of such material be required, the methods previously described (5, 6, 7, 8) may be used; but care must be taken that throughout the analysis there is a sufficient quantity of ammonium chloride present to prevent the precipitation of magnesium by ammonia.

In reporting the result of an analysis of a chalk or limestone, it is generally sufficient to state total lime as CaO, and its equivalent of $CaCO_3$ separately, MgO, loss on ignition (which includes CO_2, H_2O, and organic matter), silica, ferric oxide and alumina; and alkalies by difference.

MARLS, GAULT CLAY, CALCAREOUS SHALES.—These materials, as found in the British Isles, are intermediate in chemical composition between limestones and true clays and shales.

The method adopted for analysis must largely depend upon the particular material under examination: where the calcium carbonate is present in the proportion of 70 to 75 per cent., the processes described under Limestone (1 and following) may be used. When the $CaCO_3$ does not exceed 25 per cent., the methods to be described for clays and shales may be used, care being taken that, after treatment with sulphuric acid, sufficient 10E HCl is used to take up all the calcium sulphate formed, or this will be included erroneously in the "insoluble."

The following method giving total SiO_2, Fe_2O_3, Al_2O_3, CaO, and MgO may be used in most cases satisfactorily.

(16) Weigh into a capacious platinum crucible or

capsule 1·0 gram of the dry sample (3), and ignite in muffle at a good red heat for twenty minutes. Cool in desiccator and weigh rapidly.

Loss in weight × 100 = percentage loss on ignition.

(17) Mix the ignited residue with about 5 grams of fusion mixture or pure anhydrous sodium carbonate in a platinum crucible or large capsule, using the smooth end of a glass rod to incorporate the contents.

Heat carefully over a bunsen burner for about ten minutes, and then over a blast burner or in a muffle until the mixture is in a state of quiet, complete fusion. Rotate the crucible to spread the fused mass as much as possible, and then allow to cool rapidly by standing in a little cold water or on a cold slab. Place in a large evaporating basin, cover with distilled water and digest on hot plate until the mass has broken up and the crucible and lid can be washed clean. If necessary, a little 5E HCl may be used to ensure the crucible being clean. Then add sufficient 10E HCl, a little at a time, covering the dish as far as possible with a clock glass to prevent loss, until a clear solution is obtained.

Evaporate to dryness on the hot plate or air bath, remove and add about 10 c.c. of 10E HCl, and wash any material from the clock glass and sides down into the bottom of dish. Evaporate slowly to dryness, if necessary breaking up any lumps that form with the end of a glass rod; when dry, bake on the hot plate for at least one hour.

Remove from the hot plate and, when nearly cool, add 25 c.c. 10E HCl and sufficient water to dissolve the chlorides formed.

Decant through a 12½ cm. rapid paper, add more acid to residue in basin and warm if necessary, wash till free of chlorides.

Re-evaporate filtrate in same basin, proceed as above, filter, wash, and place both filters whilst moist in a platinum crucible, ignite, and weigh.

Weight of residue × 100 = "uncorrected SiO_2."

Moisten with distilled water and half fill crucible with HF, add 5 drops of 36E H_2SO_4, warm in fume chamber until dry, repeat, and heat to bright redness and then blast or place in muffle for five minutes. Cool in desiccator and weigh; multiply by 100 and deduct from "uncorrected SiO_2."

Residue may be analysed for Al_2O_3, CaO, and MgO, but is chiefly Al_2O_3, or it may be fused, as described above, and the solution added to main filtrate.

(17*b*) The total $Al_2O_3 + Fe_2O_3$ may be estimated in the filtrate from SiO_2 separation (17), as described in (7), but first ppt. should be filtered off, lightly washed, dissolved in hot 10E HCl, reprecipitated with 10E NH_4OH, filtered, washed, dried, ignited, and weighed in platinum crucible.

Weight of ppt. × 100 = per cent. $Fe_2O_3 \times Al_2O_3$.

(17*c*) **Estimation of FeO_2.**—Fuse the ignited ppt. from (17*b*) in the platinum crucible with pyro- or bi-sulphate of potassium, allow to cool and dissolve in cold distilled water, acidify with 5E H_2SO_4, and estimate iron volumetrically (26) or (27).

If preferred, the filtrate from (17) may be made up to a known volume and divided into equal portions, using one for determination of Fe_2O_3 (25, 26, 27).

In the filtrates from (17*b*) estimate CaO as in (8) and MgO (11).

The purity of the reagents employed should be tested by performing a "blank" analysis using same quantities.

Epitome.

Fuse 1 gram with fusion mixture.
Dissolve mass in water and hydrochloric acid.
Evaporate to dryness, bake, cool, take up with water and hydrochloric acid.
Filter, wash, and weigh SiO_2.
Ppt. $Al_2O_3Fe_2O_3$ with ammonium hydrate (7).
Filter, wash, weigh.
Estimate Fe_2O_3 (17*b*).
Filter, wash, weigh.
Ppt. CaO with ammonium oxalate (8).
Filter, wash, estimate.
Evaporate to dryness, add 30 c.c. nitric acid.
Estimate MgO as $Mg_2P_2O_7$ (11).

(18) **CLAY.**—A mechanical analysis of clay, except for brickmaking, is not often required. When necessary it may be carried out on the undried samples by elutriation. For this purpose an apparatus specially made may be used, or a series of bottles or jars can be fitted up as follows:—

Choose three wide-mouthed bottles and fit them with sound corks, each bored with two holes to carry fairly large glass tubing, in each case one piece being carried to the bottom of the vessel and the other just through the cork. The longer tube of the first bottle is connected to a water tap or reservoir, the smaller piece to the succeeding bottle, and so on, so that a stream of water may be run through the whole apparatus, the overflow from the last bottle being caught in a large jar or pail.

A weighed amount, say 100 grams, of the clay is placed in the bottle attached to the tap and a gentle stream caused to circulate through the apparatus until the overflow water comes over quite clean. The water is then turned off, and the material in the bottles allowed to subside. The bulk of the water is poured away, and the solid matter washed out into a weighed dish, dried, and weighed. The residues being reported as coarse (sand), medium, fine, and very fine by difference. All results being calculated on the dried clay. If necessary, the fineness of each portion upon standard sieves may be taken. As stated previously, however, upon a cement works using modern machinery the physical condition of the raw material is of little importance compared with the chemical composition.

The moisture should be "approximately estimated" (2), and the whole sample, when dry, powdered. A smaller sample obtained by quartering should then be dried in the air oven and kept for analysis in a weighing tube or stoppered bottle.

(18*b*) *Soluble Salts.*—Boil 5 grams of the dried clay with 250 c.c. of distilled water for half-an-hour in a hard glass flask, make up loss due to evaporation. Allow to settle, and filter, best by means of an earthenware cone and filter pump. Wash residue with hot distilled water, evaporate filtrate to dryness at 110° C., and weigh.

Residue × 20 = per cent. soluble salts.

(18*c*) In order to remove carbonates, treat 5 grams of the clay with E HCl, which also may remove colloidal hydroxides of iron and aluminium. Filter, wash, dry, and weigh residue. Estimate calcium, alumina and iron in the filtrate. Unless there is a considerable proportion of carbonate present this operation may well be omitted.

(19) *Partial Rational Analysis.*—Treat 1 gram of the dry powdered clay or residue from 18*b* and/or 18*c* in a large porcelain or platinum dish with 20 c.c. 36E H_2SO_4; rotate cautiously to break

up any lumps, cover with a clock glass, and heat very gently, best over an argand burner, for 10 hours (over night). In the morning remove the clock glass and increase the heat sufficiently to steadily drive off the sulphuric acid.

When no more fumes are evolved, place on the hot plate for about twenty minutes. Remove from the hot plate, allow to cool somewhat, and then add 25 c.c. of 10E HCl and a little distilled water.

Filter through a 12·5-cm. rapid paper, retaining the insoluble matter in the dish. Add another 25 c.c. of hydrochloric acid, digest on the hot plate for a few minutes, add water, and filter as before.

Repeat this operation, using altogether 75 c.c. of acid, then wash by decantation until free from chlorides, and when cold make filtrate up to 500 c.c. (24).

Wash any material upon the filter paper back into the dish and boil with 25 c.c. of 5E Na_2CO_3 solution or with 1 gram of sodium carbonate crystals and sufficient water, for about fifteen minutes. Filter, whilst still hot, through a 12·5-cm. rapid paper, and wash with boiling water until quite free from any trace of alkali. Reserve filtrate for SiO_2 estimation (23).

Dry residue, ignite in platinum crucible, and weigh.

Weight × 100 = per cent. insoluble, sand, etc.

(20) After weighing, treat the insoluble matter

in the platinum crucible with about 5 c.c. of hydrofluoric acid and a few drops of 36E H_2SO_4, warm over a small argand flame in a good draught cupboard until no further fumes are evolved. Repeat three times, when there should be only a small residue remaining.

Treat this residue with 10E HCl, warm in a small dish or beaker, and filter through a 7-cm. rapid paper.

(21) In the filtrate from above, estimate (7) Al_2O_3.

Weight × 100 = per cent. Al_2O_3 in insoluble.

It is usual then to calculate the Al_2O_3 to felspar $6SiO_2.Al_2O_3.K_2O$ thus:

Per cent. Al_2O_3 × 3·5 = per cent. SiO_2.

Per cent. Al_2O_3 × ·9 = per cent. K_2O.

Per cent. insoluble − felspar = per cent. quartz. (See *Appendix* for example.)

The insoluble matter can then be reported as shown in *Appendix* (1A). The result thus obtained is not absolutely accurate, but suffices for most purposes.

(22) Instead of treating the residue (19) with hydrofluoric acid, the SiO_2 and Al_2O_3 can be estimated by fusion as in (17), and for cement-making purposes this procedure is recommended.

(23) Acidify the alkaline filtrate from the insoluble estimation (19) with 10E HCl, and evaporate carefully to dryness in a platinum or porcelain

dish; bake for one hour, allow to cool, and then take up with water and a little 10E HCl, filter through a 12·5-cm. paper and wash till free from chloride. Dry, ignite in muffle for one hour, and weigh.

Weight × 100 = per cent. SiO_2.

(24) The filtrate, which has been made up to 500 c.c., is divided. In one part (250 c.c.) the $Al_2O_3 + Fe_2O_3$, CaO, and MgO are estimated as in (7), (8), and (11), (17*b*) or (17*c*).

(25) *Separate Estimation of* Fe_2O_3.—In the other part the $Al_2O_3 + Fe_2O_3$ is pptd. as in (7), filtered and washed slightly; it is then redissolved in dilute acid and the iron estimated volumetrically after reduction, using standard potassium permanganate or bichromate solution.

(26) **Estimation of Iron by means of Standard Permanganate.**—Dissolve the ppt. of $Al_2O_3 + Fe_2O_3$ in 5E H_2SO_4 and wash into an Erlenmeyer flask, add fair excess of the acid and a few small pieces of pure zinc. Fit the flask with a cork bearing a bunsen valve—*i.e.*, a piece of glass tube to which is attached a piece of rubber tubing having a longitudinal slit and closed with a glass rod or clip, and place in a slightly warm place until reduction is complete. This is approximately indicated by the solution becoming colourless, when a drop should be withdrawn on the end of

a glass rod and tested by means of ammonium or potassium sulphocyanide solution, the reduction being complete when there is no pink colouration with that reagent.

Filter the reduced iron solution rapidly and wash into a clean flask; if necessary, add more sulphuric acid; and titrate with standard permanganate [17A] solution until a permanent pink colour is obtained.

No. of c.c. Fe_2O_3 factor 200 per cent. Fe_2O_3.

Subtract from Al_2O_3, Fe_2O_3 found (24) Al_2O_3.

(27) **Estimation of Iron by means of Standard Bichromate.**—Dissolve the ppt. of $Al_2O_3 + Fe_2O_3$ in as small a quantity of 5E HCl as possible, wash into an Erlenmeyer flask fitted with bunsen valve, dilute to about 200 c.c., and add 20 c.c. of 2E Na_2SO_3. Boil for twenty minutes or until free from SO_2. Cool as quickly as possible and test as above; if reduction is complete, titrate with the standard bichromate [18A]. On a clean spotting tile have ready a number of drops of freshly prepared, very dilute potassium ferricyanide solution, run the bichromate solution, at first a few c.cs. at a time, later drop by drop, into the iron solution; after every addition abstract a drop by means of a glass rod and place in contact with the "spots" of ferricyanide. The reaction is complete when no blue or green tint is produced with the ferricyanide.

No. of c.c. × Fe_2O_3 factor × 200 = per cent. Fe_2O_3.

Epitome.

Treat 1 gram for ten hours with 36E H_2SO_4, dry and bake.
Take up with three portions 10E HCl, filter, wash.
Residue = SiO_2 and insoluble.
Boil residue with 25 c.c. 3E Na_2CO_3.
Filter, wash, residue = insoluble.
Filtrate, acidify, evaporate = SiO_2.
Filtrate from insoluble = Al_2O_3, Fe_2O_3, CaO, MgO (7, 8, 11, 17*b*, 17*c*, 25).

(28) **Loss on Ignition.**—Ignite 0·5 gram of the powdered and dry sample for twenty minutes in a muffle. Cool in desiccator and weigh rapidly.

Loss in weight × 200 = per cent. loss on ignition.

If an ultimate analysis only is required this residue (28) may be treated as (17) and the "rational" method (19) omitted.

(29) **Total Sulphur.**—One gram of the dry sample is mixed with 10 grams of a finely powdered mixture of Na_2CO_3 (10 parts) and KNO_3 (1 part) and heated to quiet fusion in a covered platinum crucible.

The fused mass is dissolved in water, acidified with 10 E HCl, and filtered, if necessary. The filtrate is boiled, and to it, whilst still gently boiling, is added 10 c.c. of E $BaCl_2$; after a few minutes the ppt. is allowed to settle in a warm place for some hours, filtered through a "sulphate" paper, washed, dried, and ignited and weighed as $BaSO_4$.

Weight of $BaSO_4$ × ·137 × 100 = per cent. sulphur.

Epitome.

Fuse with sodium carbonate and potassium nitrate.

Take up with hydrochloric acid, filter, wash.

Ppt. with barium chloride solution, weigh as $BaSO_4$.

(30) **Sulphur present as Sulphate.**—Weigh out 1 gram of the sample into an evaporating dish, add a little water and 25 c.c. of 10E HCl, warm twenty to thirty minutes, then add 10 c.c. of 10E HCl and water. Filter and wash. Ppt. with E $BaCl_2$, and treat as (29).

Weight of $BaSO_4$ × ·343 × 100 = per cent. SO_3.

If this is calculated to $CaSO_4$ its equivalent of CaO must be deducted from the amount of lime found.

Thus per cent. SO_3 × 1·7 = per cent. $CaSO_4$.

For each 1·0 per cent. $CaSO_4$ deduct ·41 per cent. from CaO.

The sulphur present as sulphate must be deducted from total sulphur found, in order to obtain sulphur present as sulphide.

(31) **Alkalies.**—Soda and potash may conveniently be estimated as follows :—

Mix in an agate mortar 1 gram of the dry clay with its own weight of pure ammonium chloride and 6 grams of pure precipitated chalk. Place the mixture in a platinum crucible and heat gently at first over a bunsen burner and then for one hour at a red heat, keeping the crucible covered ; cool, transfer the mass, which should not be fused, to an evaporating dish, and wash out the crucible with distilled water into the dish. Dilute somewhat, heat to boiling, filter, and wash into a beaker. Add 1·5 grams of solid ammonium carbonate, evaporate to about 50 c.c., add a little more carbonate + ammonia ; then filter and wash. Acidify the filtrate with 10E HCl, then evaporate to dryness in a weighed platinum dish, heat gently at first to drive off NH_4Cl and then to a dull redness ; cool in desiccator and weigh.

The weight = $KCl + NaCl$.

In order to obtain an accurate result, it is necessary to perform a "blank" experiment, and to deduct result so obtained from above.

(32) **Separation of Soda and Potash.**—Dissolve the mixed chlorides in about 5 c.c. of water and add sufficient E $PtCl_4$ to convert into the double chlorides, assuming the whole to be $NaCl$, 117 grams of which require 336·38 grams of $PtCl_4$. One c.c. of E $PtCl_4$ solution contains ·0841 gram. The mixture is then taken nearly to

dryness on the water bath and 15 c.c. of alcohol added; the dish is then allowed to stand for three hours with an occasional rotation.

When the ppt. has well settled, the clear liquid is poured off through a filter paper which has been dried and weighed, the ppt. is washed by decantation with alcohol and thus transferred to the filter paper and again washed, using a small wash bottle containing alcohol until filtrate is colourless. The paper and K_2PtCl_6 is then dried in an air or steam oven at 100° C., cooled in a desiccator, and weighed.

Weight − weight of filter paper × ·3067 = KCl.

Subtract this from weight of mixed chlorides NaCl.

$KCl \times \cdot 63204 \times 100 =$ per cent. K_2O.

$NaCl \times \cdot 53077 \times 100 =$ per cent. Na_2O.

Epitome.

(31) Fuse with ammonium chloride and calcium carbonate.
Digest with water, filter, and wash.
Add ammonium carbonate, filter, and wash.
Evaporate to dryness.
Ignite, weigh.

(32) Dissolve in water, ppt. with $PtCl_4$ and alcohol, filter.
Wash with alcohol, weigh on tared paper.

(32*b*) The proportion of potash and soda may be more simply obtained by the following method:—Dissolve the mixed and weighed chlorides in a little distilled water. Estimate the total chlorine as described under (80), but use $\frac{N}{10}$ $AgNO_3$ [19A]. Then

$$\text{Weight of K} = \frac{[(A - B) \times 1{\cdot}54] - B}{0{\cdot}63}$$

$$\text{and} \quad ,, \quad \text{Na} = \frac{B - [(A - B) \times 0{\cdot}91]}{0{\cdot}63},$$

where A = weight of mixed chlorides, and B total weight of Cl found.

$$K \times 1{\cdot}2 \times 100 = \text{per cent. } K_2O.$$
$$Na \times 1{\cdot}34 \times 100 = \quad ,, \quad Na_2O.$$

For cement-making purposes an accurate determination of K_2O and Na_2O is not often necessary.

Shales, Sandstones, and Slates.—The methods adopted for the analysis of these materials must depend upon circumstances. As a rule, the following processes, already fully described, will be found suitable:—

Moisture (3), loss on ignition (4), followed by fusion (17) and estimation of the SiO_2 (17), Al_2O_3 Fe_2O_3 (7), CaO (8) and MgO (11).

Natural Cement Rock.—This may generally be treated as a limestone, carrying out the following estimations:—

Loss on ignition (4), silica and insoluble (5 and 6) or a fusion of the insoluble may be made (17), Al_2O_3 Fe_2O_3 (7), CaO (8 and 9), magnesia (11), CO_2 (14 or 15) if necessary or $CaCO_3$ and $MgCO_3$ may be obtained by calculation; sulphates (13) and sulphides (29).

(33) **Slags.**—Weigh out one gram of the finely powdered sample into a platinum crucible and fuse with 3 grams of fusion mixture; separate the SiO_2, as in (17).

(34) The filtrate from the silica is warmed in a capacious beaker; a little solid and then 5E Am_2CO_3 solution added until a ppt. just forms. Then add a drop or two of 5E Acetic acid and excess of 4E Sodium acetate solution. Boil gently for ten minutes and filter, whilst still hot, through a 15-cm. rapid paper; wash.

The filtrate must be clear and colourless, and is used for manganese estimation (36).

(35) Dissolve the pptd. basic acetates of iron and alumina in 10E HCl, and make up to a known volume with cold distilled water.

Divide this solution into two equal parts, in one estimate total $Fe_2O_3 + Al_2O_3 + P_2O_5$.

Use the other portion for the estimation of Fe_2O_3 by means of standard permanganate (26) or standard bichromate (27).

The Fe_2O_3 found must be deducted from the total $Fe_2O_3 + Al_2O_3 + P_2O_5$ in order to obtain per cent. of $Al_2O_3 + P_2O_5$.

The Fe_2O_3 should then be calculated to FeO and reported as such.

$$Fe_2O_3 \times 0{\cdot}9 = FeO.$$

(36) Concentrate the filtrate from (34) if necessary, allow to cool, add 3 c.c. of bromine, stir, and add carefully 20 c.c. of 20E NH_4OH, boil for twenty minutes, filter, wash, ignite, and weigh as Mn_3O_4.

Weight $\times$ 0·93 $\times$ 100 = per cent. MnO.

(37) Boil the filtrate from (36), add sufficient ammonium oxalate solution to ppt. lime and filter and estimate as in (8) and (8*a*).

(38) Evaporate filtrate from (37) to dryness and estimate magnesia (11).

Epitome (33-38).

Fuse with fusion mixture.
Estimate SiO_2 (17).
Separate iron and alumina, etc., as basic acetate (34).
Estimate iron volumetrically.
Manganese with bromine water and ammonia.
Lime and magnesia in filtrate.

(39) **Phosphorus and Sulphur.**—Fuse 2 grams at least of the sample with sodium carbonate

and potassium nitrate, treat the melt with hot water until it disintegrates, boil, filter off the bases, and wash well. Allow the filtrate to cool and make up to 500 c.c. Divide into two portions (A) and (B). Acidify A with HNO_3 and add a large excess of ammonium molybdate solution, allow to stand for twelve hours, at about 50° C.; if the temperature is allowed to rise, any arsenic present will also be pptd. Pour off the clear liquid through a "sulphate" paper, and test with a little more molybdate solution to ascertain whether all phosphate has been pptd.; if so, filter and wash any ppt. adhering to beaker with E HNO_3. Then, using a fresh beaker to collect the filtrate, dissolve the ppt. in 5E NH_4OH, rinse out the beaker, and wash the filter paper well.

Add excess of magnesia mixture, treat as in (11).

Weight of $Mg_2P_2O_7 \times \cdot 638 \times 100$ per cent. P_2O_5.

Epitome.

Fuse with sodium carbonate and potassium nitrate.

Dissolve in water, acidify with nitric acid, ppt. with ammonium molybdate, filter, dissolve in ammonia, ppt. with magnesia mixture.

Subtract from $Al_2O_3 + P_2O_5$ and Fe_2O_3 (35).

(40) **Sulphur.**—Acidify (B) with 10E HCl, add 10 c.c. E $BaCl_2$ solution, treat as in (29).

$BaSO_4 \times \cdot 137 \times 100 =$ per cent. sulphur.

CHAPTER III.

CALCULATION OF PROPORTION OF RAW MATERIALS.

FROM the analysis of any given material it is possible to estimate its usefulness for cement making within certain limits. With entirely untried materials actual tests on as large a scale as possible should be carried out.

Any of the larger English and American text-books on cement manufacture contain full information as to the ideal composition.

LIMESTONES.—Unless a calcareous clay or shale is to be used, the $CaCO_3$ content must reach 75 per cent. A poor limestone may sometimes be enriched by using a purer stone to raise the percentage of $CaCO_3$. About 2 per cent. MgO renders the stone useless for cement making to standard specification; it is desirable that only 1 per cent. or less be present.

For use with rotary kilns, a little sulphur as sulphide or sulphate is immaterial either in the stone or clay.

CLAY, SHALES, ETC.—The proportion of SiO_2 to $Al_2O_3 + Fe_2O_3$ should be $2\frac{1}{2}$ or 3 to 1.

That is, per cent. SiO_2 should equal per cent. $R_2O_3 \times 3$, unless working with a siliceous limestone or a stone of nearly correct proportions which is only deficient in one constituent.

A clay containing nodules of iron pyrites should be avoided, as a source of possible, if not probable, trouble.

Calculation from Formulæ.—Several formulæ exist whereby the proportions may be calculated in which raw materials should be mixed, but even the best of these only serve as a rough guide in actual practice. From experience the most favourable data to work upon is the percentage of $CaCO_3$, as this can be readily checked and altered. The percentage of $CaCO_3$ in a raw mixture never varies greatly from 75 per cent.

In order to obtain approximately the correct proportions, proceed as follows. For example, see *Appendix* (3A).

(1) From per cent. $CaCO_3$ in limestone deduct 75 or per cent. required in mixture; the result should be the weight of clay or shale required.

(2) From 75 or per cent. $CaCO_3$ required in mixture deduct per cent. $CaCO_3$ in clay or shale; the result should be weight of limestone required.

Of course, the weights so obtained can be read as grams, lbs., cwts., tons, or any unit required. In each case, when using raw damp material, the moisture must be estimated and allowed for, as obviously more will be required than when using perfectly dry stone or clay.

As a result of researches into the composition of an ideal Portland cement, various experimenters have put forward formulæ for preparing the raw mixture when the composition of the materials is known. The following will be found a good working formula :

(1) Multiply per cent. SiO_2 in limestone by 2·8 ; multiply per cent. Al_2O_3 in limestone by 1·1, and add the products.

(2) Deduct result from per cent. CaO in stone = per cent. CaO available for combination with clay (y).

(3) Per cent. SiO_2 in clay × 2·8 + Al_2O_3 in clay 1·1, gives CaO required by 100 parts of clay.

Deduct per cent. CaO in clay and remainder = amount required to be added (x).

As the available CaO (2) in 100 parts of limestone is known, by simple proportion, parts of limestone to be added can easily be calculated. Thus let x = amount of CaO required for 100 parts of clay, and y = per cent. available in limestone ;

then $\frac{x \times 100}{y}$ = parts of stone to each 100 parts of clay.

For examples worked out, see *Appendix* (2A).

ANALYSIS OF SLURRY AND OTHER RAW MIXTURES.

SLURRY.—In order to obtain an average sample of the slurry being washed, the constant supervision of the chemist or of a trustworthy assistant is absolutely essential. Owing to different prevailing conditions, it is impossible to lay down any fixed plan of sampling. As a rule, samples should be taken at regular short intervals as the slurry leaves the grinding plant; these should, at longer intervals, be thoroughly mixed and a smaller average sample taken for examination.

For sampling slurry-mixing tanks or silos various devices exist, one of the simplest, perhaps, being a tin can with a perforated lid: the can is weighted at the bottom and attached at the lid to a long pole or piece of cord. The body of the can is fitted to the lid and held firmly by a simple bayonet clip or in the same way that incandescent electric lamps are fitted to their holders.

The vessel is lowered into the tank at different depths and then slowly raised; as it is withdrawn the semi-liquid rushes in through the perforations and a sample is thus obtained. The instrument sometimes used for tar sampling will also be found efficient. More elaborate apparatus will be found figured in many English and American works on cement manufacture. A single sample taken at one depth should never be used for analysis. A "grain sampler" will be found very useful for sampling dry raw meal or cement.

Samples of slurry that have been allowed to stand, even a short time, must be well shaken before examination.

(41) **Moisture.**—Into a dry and tared flat-bottomed porcelain dish weigh out 5 grams of the well-mixed slurry, place in a hot air oven or on a hot plate at about 110° to 120° C. until quite dry, cool in a desiccator and weigh rapidly.

Loss in weight × 20 = per cent. moisture.

(42) Remove the dried material from the dish with a spatula, powder finely in an agate mortar, and place in a stoppered weighing tube. Dried slurry very quickly absorbs moisture, so it is better to place the powdered material in the oven for a short time before bottling.

(43) **Analysis.**—Ignite 0·5 gram in a platinum crucible in the muffle for fifteen to twenty minutes at a good red heat. Cool in desiccator and weigh.

Loss in weight × 200 = loss on ignition.

(44) Upon the ignited sample carry out the analysis as described under Limestone, SiO_2 and Insol. (5 and 6); Insol. (6); $Al_2O_3 + Fe_2O_3$ (7); CaO (8); and MgO (11); CO_2 (14) or (15); sulphur and sulphates (13); and (29) if necessary.

(45) The excess of the loss on ignition over the CO_2 estimated or found by calculation from CaO and MgO may be stated as "organic matter" without any grave inaccuracy.

For works routine the following determinations are of great importance. Moisture (as in 41).

(46) "**Fineness.**"—Weigh out 100 grams of the wet slurry with fair accuracy, wash it with a gentle stream of water from the tap into a 180- or 200-mesh sieve especially kept for the purpose. Continue the washing until the water that runs through is quite clear. Then wash the residue up together, and with a wash bottle transfer it to a small evaporating dish; place upon the hot plate or in an oven, and dry.

Weigh the residue; calculate the percentage upon the dried material thus:

$$\frac{\text{Weight of residue} \times 100}{(100 - \text{per cent. moisture})} \text{ per cent. fineness.}$$

With dry meal sift 100 grams in the usual way, and weigh the residue:

$$\text{Weight} = \text{per cent. residue.}$$

It is sometimes useful to estimate the chalk in this residue by a rapid method.

(47) **Estimation of $CaCO_3$.**—In most works in England this constitutes the greater part of the daily routine, and as the production of a uniform article largely depends upon the use of a

regular raw mixture, too much time and attention cannot well be paid to this important item.

Owing to the use of calcimeters, the actual routine determinations can generally be safely carried out by untrained assistants who by continual practice have become proficient in this part of the laboratory work. Constant supervision and occasional "check" estimations by the chemist are necessary with even the most conscientious workers, in order to keep them up to the required standard of speedy accuracy.

There are many good calcimeters on the market, all more or less inaccurate: but when once the error is known, if it be constant, the actual accuracy of the result is practically unimportant, as the chemist in charge should know at what figure to work with any given apparatus. Where more than one machine is in constant use, they should be so regulated as to give strictly comparable results. For this purpose it is better to prepare a standard raw mixture by careful and accurate weighing of the raw materials after analysis than to use powdered and dry calcite.

(48) **Preparation of a Standard Dry Slurry or Raw Meal.**—Carefully analyse a good sample of the ordinary mixture that is known to produce the best results in practice. Then dry a small sample of each of the raw materials, reduce to the necessary fineness, and accurately weigh up and

carefully mix in the proportions found by analysis of raw materials and slurry.

A stoppered bottle of this should always be kept ready for use. When a check analysis is required, it is only necessary to dry a little of the standard slurry in the air oven at 105° C., and allow it to cool in the desiccator.

(49) Calcimeters may also be standardised, using dry pure calcite which has been powdered in an agate mortar. With calcimeters upon which it is usual to take ·5 gram of slurry, it is convenient to take only ·375 gram of calcite, which should give a result equivalent to 75 per cent. $CaCO_3$ after the usual corrections.

(50) **Slater's Calcimeter.**—This is an instrument which deserves to be more widely known and used than at present. It is fairly simple in construction, and can readily be adjusted. It requires perhaps rather more skilful manipulation than some others, owing to the necessary alterations of the amounts used at varying temperatures.

As shown in the illustration, it consists mainly of two parts, the outer containing vessel A, and the inner bulb and tube B. Upon the leg of this bulb are graduations usually running by 0·5 from 70 to 80, which are read direct as percentages of $CaCO_3$, as described later.

Above the bulb is a zero mark to indicate the correct amount of paraffin with which the instrument has to be filled before use. In the bulb a regular raw mixture, too much time and attention cannot well be paid to this important item.

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The instrument as sent out generally requires standardisation (see below). It is used as follows:

(1) Take the reading of the barometer in millimetres, and find the nearest corresponding mark on the float.

(2) Take the temperature of the oil in degrees C.

(3) From a table ascertain the correct weight of material to use in milligrams, and weigh this out accurately.

(4) Transfer the weighed slurry to the generator bottle (D), which must be quite dry.

(5) Run from a burette 5 c.c. of dilute commercial HCl (1 : 1) into the test tube, attached to the generator bottle stopper, by means of the hole in the side.

(6) Fit the leading tube from calcimeter to the bottle, and slip the small piece of rubber over the outlet hole.

(7) Tip the acid on to the slurry and shake well to disengage CO_2. The float will then sink in the paraffin, and the mark on the stem which is found to correspond with the correct barometer reading on the float should be the percentage of $CaCO_3$ in the slurry or material taken.

By making use of tables similar to those in the Appendix materials higher in $CaCO_3$ than 80 per cent. may be estimated (28A).

The following precautions should be observed in using this apparatus:

(1) Avoid holding the generator in the hand

longer than is absolutely necessary, and hold the neck and stopper of the bottle.

(2) See that the rubber is over the outlet hole before spilling the acid, and that it is removed before disconnecting generator.

(3) Spill the acid carefully so that none be forced up the leading tube.

Use paraffin that has been placed over fused calcium chloride, and has such a specific gravity that the hydrometer sinks to the red mark on its stem. Use acid of the correct strength.

Standardisation of Slater's Calcimeter.—Follow the appended instructions for using the calcimeter, but take a known weight, say 1 gram, of a carefully prepared standard slurry (48), and note the reading which should show 75 per cent. $CaCO_3$, or as the case may be. If the reading is too high, as it probably will be, calculate the reduction in the weight necessary to attain a correct reading, and repeat the operation until a correct result is obtained. This should be done through a good range of temperature, so that a complete table may be drawn up by interpolation. Once having been correctly standardised, any slight adjustment found necessary may be made by adding or removing a little oil.

This instrument, and a specially prepared set of weights for use therewith, may be obtained from Messrs. Baird & Tatlock (London), Ltd.

The Schleibler, Faija, and other calcimeters are so well known and have been so frequently described and illustrated as not to warrant description here. A modification of Schleibler's instrument introduced by Mr. H. K. G. Bamber, F.C.S., and much used in the A.P.C.M. Works, will be briefly described and illustrated (Fig. 4).

(51) **Bamber's Calcimeter.**—Weigh out accurately 0·5 gram of the dried and finely pulverised slurry and transfer to generator bottle (14). Run into the gutta-percha tube 8 c.c. of hydrochloric acid (sp. gr. 1·125) and place carefully in bottle. Fill measuring tubes with water from Woulff's bottle (*b*), or reservoir, until water stands at zero mark on the graduated tube.

Fix stopper to generator bottle and immerse to the neck in the running water which fills the lead-lined wooden vat, until it is at the same temperature as the water in the tubes 2 and 3. This is so when, upon opening clip (13), the water in the tubes remains at the same level.

Take the generator bottle in the right hand and cautiously spill the acid in the tube upon the slurry. At the same time with the left hand release clip (7), shake the bottle vigorously for about half a minute, and replace under water in the nest. During the evolution of the gas care must be taken to keep the level of the water in the plain tube (3) about an inch above that in the measuring tube (2). Allow to cool for about three minutes, adjust levels of water, and take reading. Note temperature of water and also barometric pressure.

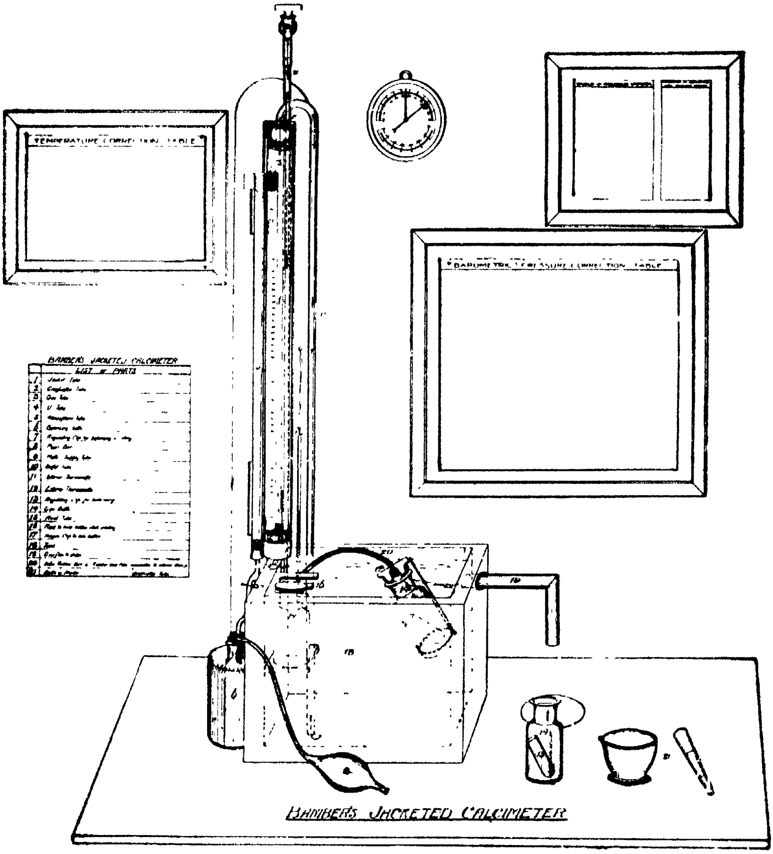
TEMPERATURE CORRECTION TABLE
BAROMETRIC PRESSURE CORRECTION TABLE
BAMBER'S JACKETED CALCIMETER
LIST OF PARTS
BAMBER'S JACKETED CALCIMETER

Fig. 4.

To calculate Percentage of $CaCO_3$.—To calcimeter reading in c.c. add correction for absorption of CO_2 by acid. This may be obtained by performing a check estimation, using Iceland spar or standard slurry. The figure usually taken for this instrument lies between 3·5 and 4·2 c.c. By means of table (30A)—*Pressure of aqueous vapour* in *Appendix*—note deduction to be made from observed barometric pressure at existing temperature. Refer to table (31A), and under the ascertained temperature find the corrected calcimeter reading (or nearest): in a line with this, denoted by heavy type, will be found the reading at 0° C. The figure so obtained is now traced on table (32A) under the correct barometric pressure, and in a horizontal line will be found in heavy type the percentage of $CaCO_3$ at 0° C. and 760 mm. pressure.

By means of table (29A) having performed a "loss on ignition," the calculated $CaCO_3$ (54) may be obtained.

(52) Estimation of $CaCO_3$ by means of Standard Acid and Alkali.

—The following is an epitome of a method described by R. K. Meade. (*Portland Cement*, p. 231):

Prepare $\frac{2}{5}$ N HCl and $\frac{2}{5}$ N NaOH, and standardise against a standard sample of slurry. The method of standardisation and working is as follows:

Weigh 1 gram of sample into a 600 c.c. Erlenmeyer flask and run in 50 c.c. of the standard acid.

Close the flask with a cork bearing a piece of glass tube 30 in. long and ⅜ in. diameter, which acts as an air condenser. Heat the flask until steam just begins to issue from the upper end of tube. This should take about two minutes. Remove from heat and rinse the tube down carefully with water, remove cork and wash down sides of flask. Add a drop or two of 1 in 1,000 phenolphthalein or methyl orange and titrate with standard alkali until just neutral.

If the standard sample contains *L* per cent. of $CaCO_3$ and *d* c.c. of alkali are required, then to find percentage of $CaCO_3$ in other samples it is only necessary to subtract the number of c.c. of alkali required in their case from *d*, multiply by two, and add to *L*; if number of c.c. is greater than *d*, subtract *d* from that number, multiply by two, and subtract from *L*. Each c.c. of exactly $\frac{N}{5}$ N alkali is equivalent to ·02 gram or 2 per cent. $CaCO_3$; so that, after standardisation, a table may be prepared showing percentages of $CaCO_3$ corresponding to different quantities of alkali. It is necessary to prepare samples to same state of fineness for each determination.

(53) **Loss on Ignition, Calculated $CaCO_3$ and CaO in Slurry.**—When using materials containing organic matter, such as peat, etc., it will be found very useful to ascertain the $CaCO_3$ in the meal after allowing for the organic matter present, and this may be done as follows:—Find the loss

upon ignition of 0·5 gram dry slurry or meal as in (43).

Loss in weight × 200 = per cent. CO_2 + H_2O and organic matter (A).

(54) **Calculated $CaCO_3$.** — Calculate the $CaCO_3$, found upon calcimeter, into CO_2 by dividing thus,

$$CaCO_3 \times 0{\cdot}44 = \text{per cent. } CO_2 \text{ (B)}.$$

Then,

Loss upon ignition (A) − CO_2 (B) = organic matter (C).

And 100 − organic matter (C) = parts of material in which the $CaCO_3$ exists after removing the organic matter by ignition (D).

Thus,

$$\frac{\text{per cent. } CaCO_3 \text{ found} \times 100}{D} = \text{calculated } CaCO_3.$$

The actual calculations only take a few seconds if the tables in *Appendix* be used.

(55) The residue obtained after ignition should be reserved and the CaO present determined by the rapid method as described in Chapter V. (132); after allowing for the loss upon ignition the CaO found should be approximately the same as the clinker made from this slurry will contain. Owing to various circumstances, this is not always quite

the case in practice, but this serves as an excellent check upon the calcimeter or other determinations.

The calculation is as follows:

$$\frac{\text{Per cent. lime in residue} \times 100}{(100 - \text{loss})} = \text{calculated CaO.}$$

(56) **Control and Alteration of the Raw Mixture.**—Owing to the various systems in vogue on different works, it is impossible to lay down any hard and fast rules for the control and regulation of the slurry or raw meal. The growth of the cement industry in this and other countries has brought about the successful use of raw materials differing so widely in composition as to require in some cases plant and methods of working of quite a distinct and special character. Before finally deciding upon a method of working, the chemist in charge should assure himself that the routine proposed fulfils the following requirements:

(1) That there is a regular supply of raw material sufficient to keep the mills and kilns going continuously.

(2) That the unit loads of materials are of constant weight or bulk.

(3) That it is possible to readily obtain a sample of either raw material being used at any moment for check analysis.

(4) That accurate returns of the quantity of material used be sent to the laboratory at stated periods, in order that any deviation from the

working instructions may be at once noted. It is absolutely essential that a responsible person be in charge of each mill and be made answerable for any irregularity of working either in the feeding or output.

The importance of being able to obtain an average sample of the slurry or raw meal for routine analysis has already been mentioned. In order that this work be not interfered with, the samples should be fetched by a laboratory assistant, marked for reference, and immediately examined. All results should be carefully entered up for future reference.

Before making an alteration in the raw supply at any mill, the cause of the erratic behaviour should, as far as possible, be ascertained and noted; then the desirability of making a temporary or permanent alteration will at once be known and acted upon. Time should be given for the alteration to have effect before again checking the output. It is unwise to irritate the mill hands by useless and vexatious alterations and orders.

In working upon the dry system it is best to keep a silo full of limestone or clay, and work so as always to require adjustment in one direction. For this purpose, either wet or dry process, it is well to have three tanks or silos for the raw meal: one to run the make into, one that may be tested and corrected, and one containing material of the correct composition. It should be impossible for the kiln attendants to use any material but that passed for use by the chemist in charge ; even then the meal, as fed into the kilns, should be regularly sampled, checked, and the results entered in the laboratory records. In a word, too much care cannot well be given to this branch of the laboratory work.

CHAPTER IV.

ANALYSIS OF FUEL, LUBRICANTS, WATER, AND KILN GASES.

THE fuel used chiefly on a cement works is coal or coke. Brief descriptions only are given of the chief methods of analysis; for fuller description, especially of the calorific value determination, the inexperienced reader is recommended to consult larger works.

(57) **COAL.**—In order to obtain an average sample of the fuel, it is best to have a part of the freight set apart as it is being unloaded, say one barrow or grab full in ten, then have this well mixed and reduced to a convenient bulk by the method of quartering. When sampling a large cargo it will be found advisable to obtain several samples and carry out check assays.

When not too large, the whole of the sample brought to the laboratory should be coarsely powdered and quartered, and the portion selected for analysis all ground to pass at least the 90-in. mesh sieve must be reserved in a well-stoppered bottle or jar.

(58) **Moisture.**—It is well to estimate the moisture as soon after receiving the sample as possible.

Weigh into a weighed platinum capsule 0·5 or 1 gram of the powdered coal and heat in an air oven at 105° C. for not more than one hour. Cool in a desiccator and weigh rapidly, as dry coal is very hygroscopic.

Loss in weight × 200 (or 100) = per cent. moisture at 105°.

Epitome.

0·5 gram in oven at 105° C. for one hour.

(59) **Ash.**—Gently ignite the dried coal over a bunsen burner or in the muffle, first at a low temperature, until all the carbonaceous matter has burnt off. The colour of the ash sometimes indicates its nature.

Cool in a desiccator and weigh.

Total weight − weight of capsule × 200 = per cent. ash.

Epitome.

0·5 gram ignited in muffle.

(60) **Volatile Matter.**—One gram of the sample is placed in a large weighed platinum crucible having a well-fitting cover. Heat the crucible with lid on for two minutes over a bunsen burner, or until no more inflammable vapours are emitted, and then for a further period of three minutes

over a blast burner. Allow to cool in a desiccator, and weigh.

Loss in weight × 100 = volatile matter + moisture per cent.

Epitome.

> One gram heated in closed crucible for two minutes over bunsen, + three minutes over blast burner.

In order to obtain concordant and comparable results, it is necessary always to perform this operation in exactly the same manner.

The per cent. moisture (56) subtracted from volatile matter + moisture will give the volatile matter.

(61) "**Fixed carbon,**" or better, fixed carbonaceous residue. This is obtained by difference.

100 − per cent. (moisture + volatile matter ash) "fixed carbon."

(62) **Coke.**—This is not, as a rule, of great importance. The residue left after the determination of the volatile matter is, roughly speaking, coke. It should be tested to see whether it is friable or compact.

Coke may be more accurately determined by placing 1 gram in a covered porcelain crucible placed in a large Battersea round crucible and surrounded and packed well in and covered with powdered charcoal.

The lid of the large pot is luted on with clay, and the whole dried and heated in a wind or gas furnace until it is thoroughly hot.

After being allowed to cool, the porcelain crucible and lid are carefully removed, and the coke brushed out on to a watch glass and weighed.

Weight of coke 100 = per cent. coke.

Epitome.

One gram heated in covered crucible in furnace.

(63) **Sulphur.**—Mix 1 or 2 grams of the finely-powdered coal with twice its weight of a mixture consisting of 1 part dry K_2CO_3 and 2 parts MgO in a capacious platinum crucible and cover with a little more of the fusion mixture. Place the crucible in a hole in a sheet of asbestos board and heat over a blast burner for half an hour, or use an oil or spirit blast lamp in order to avoid contamination with sulphur from the coal gas. Allow to cool, dissolve and acidulate with E HCl, evaporate to dryness, take up with 10 c.c. of 10E HCl and a little water, filter off SiO_2 ; dilute filtrate somewhat, and estimate S as $BaSO_4$ (13). If a gas muffle is employed a blank analysis should be carried out, and the result of analysis corrected accordingly.

Epitome.

Fuse 1 or 2 grams with K_2CO_3 and MgO mixture.

Dissolve in hydrochloric acid, remove SiO_2.

Ppt. with barium chloride solution (13).

(64) **Analysis of Ash.**—Ignite 10 grams or more of the fuel in a platinum capsule until free from carbonaceous material. Use of this 1 gram or more, and determine SiO_2, Al_2O_3, Fe_2O_3, CaO, and MgO after fusion, as in (17) ; and, if necessary, upon other portions SO_3 (30) and P_2O_5 (39).

If an ultimate analysis of the coal is required, it will be necessary to fit up a combustion apparatus. In addition to the absorption apparatus, as described and illustrated in (14), a furnace, combustion tube and cylinder of oxygen will be required. The combustion tube is prepared as for use in organic analysis with copper oxide, the coal being burnt in a porcelain boat.

The nitrogen may be estimated by a modified Kjeldalh method. As an ultimate analysis is rarely required for commercial purposes, a fuller description is not necessary here.

(65) **ANALYSIS OF COKE.**—The methods described under Coal may be used for the determination of moisture (58), ash (59), volatile matter and fixed carbon (60) and (61), and sulphur (63).

(66) **CALORIFIC POWER OF FUEL.**—For this determination a calorimeter is required. One of the simplest is Lewis Thompson's ; but one of the improved forms of this instrument using oxygen gas and having electric ignition is more accurate and satisfactory.

In the ordinary form a known weight (2 grams) of the fuel is mixed with sufficient finely-powdered ignition mixture ($KClO_3$ 3 parts, KNO_3 1 part), placed in the copper cylinder and ignited, when all is ready, by means of a fuse.

The cylinder and attachment are immediately plunged into 2,000 c.c. of water at a known temperature contained in the special vessel. After combustion the increase of temperature is noted. For details consult Beringer, Phillips, or the instructions given with the instrument.

In the improved forms the coal is made into a pellet and ignited whilst under the water by means of an electric current.

The improved form of Roland Wild calorimeter with electric ignition will be found a very satisfactory instrument for use in a cement works laboratory.

1,000 grams of water, less the water equivalent of the apparatus which is stated for each instrument, are placed in the water vessel and the temperature carefully noted. 0·73 gram of the powdered and dried fuel is mixed with from 12 to 14·5 grams of sodium peroxide, and placed in the special crucible provided. The crucible is screwed to the cap, great care being taken that the rubber washer is in good condition, making a sound water-tight joint. This point is very important, as otherwise a disastrous explosion, due to the peroxide coming into contact with water, will result.

The thermometer is then fixed on the cover, and the directions given with each apparatus closely followed.

The difference between the temperature of water before test and the highest degree noted due to combustion of the fuel multiplied by 1,000 the calorific power in British thermal units per lb. of fuel.

After the determination the apparatus must be carefully cleaned, washed, and dried.

If the result is required in "gram-calories," a Centigrade thermometer is used or the B.Th.U. 252.

Whatever instrument is used, the result may be expressed in calories or British thermal units.

The "gram-calorie" is the amount of heat required to raise 1 gram of water through 1° C. The large or "kilogram-calorie" is the amount of heat required to raise 1 kilogram of water through 1° C.

The British thermal unit is stated in lbs. of water raised through 1° F. by 1 lb. of fuel.

The evaporative power is the amount of water which 1 lb. of fuel will evaporate at boiling point under normal conditions.

$$\text{Gram-calories} \times 0{\cdot}00397 = \text{B.Th.U.}$$

Determination of Heating Power of Coal and Coke.—A simple, but only approximate, method for determining the heating power of coal or coke may be carried out as follows:—Mix 1 gram of the finely-powdered dry coal with 30 grams of litharge in a No. 3 Hessian crucible, cover the mixture with a further 20 grams of litharge, and heat crucible and contents in a gas or wind furnace for fifteen minutes.

Remove from furnace, and when cold remove button of lead, clean and weigh.

One part by weight of carbon reduces 34 parts by weight of lead. One kilo of carbon when completely burnt is equivalent to 8,140 calories.

$$\therefore \quad 1 \text{ part of lead} \quad \frac{8,140}{34}, \text{ or } 239, \text{ calories.}$$

$$\frac{8,140}{34} \times \text{weight of lead reduced} = \text{calories per kilo.}$$

For use in rotary kilns the character of the coal when burning is of greater importance than the actual calorific power.

(67) **Fineness.**—This is determined in the same way as with cement, the 100-mesh sieve being used. To obtain good results in a rotary kiln the residue should not exceed 5 per cent., the coal should be quite dry and low in ash, and easily combustible.

The methods given above can be used for any class of coal, but it is desirable to use a rather higher temperature or heat for a longer period to estimate the volatile matter in anthracite or steam coal.

OILS AND LUBRICANTS. — The chief methods of examination only can be mentioned.

If much important work has to be done the chemist is recommended to consult Archbutt and Deeley's or other large work.

Lubricants may be very roughly divided into three classes—solid, semi-solid, and liquid. Only grease, fats, or oils will be here dealt with, but it may be mentioned that graphite, mineral, and compound lubricants should be free from grit, acid, or alkaline bodies or substances likely to decompose and produce acids, etc.

Before reporting on a lubricant it is necessary to know for what purpose it is intended to be used. The following tests may be considered essential:

(68) **Loss or Gain in Weight on Exposure.**—Into a weighed watch-glass place 1 gram of the oil, and expose in an air or water bath at 100° C. for twelve hours. Allow to cool in a desiccator and weigh.

Loss (or gain) in weight × 100 percentage loss (or gain).

Good mineral oils rarely lose more than 1 per cent.; some vegetable oils increase in weight owing to oxidation. The residue should not exhibit any sticky or gummy properties.

The residue may be ignited for "Ash," which should not exceed traces only.

Epitome.

1 gram at 100° for twelve hours.

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OILS AND LUBRICANTS. — The chief methods of examination only can be mentioned.

work the apparent sp. gr. at T should be corrected, as follows :—

$$\text{Sp. gr. at } \frac{T^\circ\text{ C.}}{T^\circ\text{ C.}} \times \frac{\text{Density of water at } T^\circ\text{ C.}}{\text{Density of water at } 15{\cdot}5^\circ\text{ C.}}$$

In order to avoid these calculations, the sp. gr. should be determined at 15·5° C. whenever possible.

For very viscous oils it will be found convenient to use the form of bottle that is used for testing syrups, etc.

(70) **Viscosity.**—This is best determined in a standardised Redwood viscosimeter. The viscosity of an oil varies with the temperature at which it is determined. The viscosity should be determined at various temperatures according to the purpose for which it is required. In order to obtain the viscosity of an oil, it is heated in an air bath to the required temperature, and poured into the apparatus up to the point of the gauge. The outer vessel, which may be filled with water, oil, or any other convenient liquid, is then warmed until both it and the oil to be tested are at the required temperature. A plug is then removed and exactly 50 c.c. of oil allowed to run out, and the time taken in seconds noted by means of a stop watch. For comparative results consult tables published by Redwood and others.

In order to obtain comparable and reliable results, several tests should be carried out under exactly similar conditions.

In the absence of a standardised viscosimeter, comparative tests may be carried out by using a pipette or burette and noting the number of seconds that a known volume of the oil takes to run out.

Viscosimeters are generally standardised with pure rape oil or distilled water.

In order to obtain comparable results, experiments must be carried out under exactly similar conditions, great attention being paid to the temperature both of the oil and the surrounding atmosphere.

(71) **Flash Point.** This may be readily ascertained with Abel's flash point apparatus. With a little practice the principles of the determination can soon be mastered. Full instructions are generally sold with the apparatus, or can be found in the Petroleum Acts or any work on oil analysis. For oils flashing above 100° C. the air bath is heated by means of a small bunsen burner or spirit lamp. The flash point of an oil should be higher than the temperature that will be obtained during work.

(71*a*) The "open cup" flash point of an oil may be ascertained by the following method:—

Place a suitable nickel or porcelain crucible in a hole in a sheet of asbestos board supported on a tripod stand. Suspend a thermometer over the crucible so that the mercury bulb is about half an inch from the bottom, but is well immersed in the oil, which should nearly fill the crucible.

Heat the crucible by means of a small bunsen flame so that the temperature rises gradually and evenly.

A small flame, obtained by attaching an ordinary mouth blowpipe by means of rubber tubing to another gas supply, is then passed across the surface of the oil at regular intervals until the vapours are seen to "flash." The lowest temperature at which this occurs is the flash point. The temperature at which the vapour flashes and continues to burn is known as the fire point.

(72) **Free Mineral Acids.**—Shake up 50 grams of the oil with sufficient distilled water in a stoppered flask or bottle, and allow to stand for some time; then separate the oil by means of a separating funnel. To the aqueous extract add a drop of methyl orange solution; a pink colouration will indicate the presence of free mineral acid. If sufficient in amount, titrate back to the neutral tint with $\frac{N}{10}$ or $\frac{N}{100}$ NaOH. The nature of the acid must be determined by a qualitative analysis of the extract. H_2SO_4 is the acid most frequently found.

Should the methyl orange remain neutral or indicate the presence of free alkali, repeat the test, using phenolphthalein, and titrate back with standard acid.

Epitome.

Agitate 50 grams of oil with distilled water.
Separate, add methyl orange, and titrate with
$\frac{N}{10}$ or $\frac{N}{100}$ NaOH.

(73) **Free Fatty Acid.**—Weigh out 10 grams of the oil into a flask, and add 50 c.c. industrial methylated spirit rendered just slightly pink by the addition of a drop or two of phenolphthalein and one drop of E NaOH. Shake up together well. If free fatty acid be present the solution will become colourless. Heat on a steam bath under a reflux or air condenser for half an hour, allow to cool, and titrate with standard $\frac{N}{10}$ NaOH until a permanent pink colouration is obtained. Calculate the "acid number" as milligrams of potassium hydroxide neutralised by 1 gram of oil, thus:

$$0{\cdot}56 \times \text{No. of c.c. } \frac{N}{10} \text{ alkali used} = \text{acid number.}$$

A blank experiment should be carried out in order to check result.

Epitome.

Warm 10 grams of oil with alcohol and phenolphthalein solution.
Titrate with standard alkali.

(74) **Saponification Number.**—Weigh 2 grams of the oil into a clean, dry conical flask,

add 25 c.c. or excess of semi-normal alcoholic potash solution, connect with a reflux air condenser, which consists simply of a straight glass tube about 30 inches long, and heat on the water bath for one hour, occasionally rotating the flask. Allow to cool and titrate with semi-normal acid, using phenolphthalein as indicator.

Run a blank experiment under exactly similar conditions to ascertain the number of c.c. of alkali neutralised by the oil. Calculate the saponification number as milligrams of potassium hydroxide to 1 gram of oil.

Acid used by "blank"—acid used in experiment 14 saponification number.

(74*b*) **Unsaponifiable Matter.**—Separation of fatty and mineral oils. Saponify 5 grams of the oil as in (74), using an excess of alcoholic caustic potash (50 c.c.). Reverse or remove the condenser and distil off the alcohol as completely as possible. Dissolve the soap in about 50 c.c. of hot distilled water, transfer to a separating funnel, cool, and shake up with two separate portions of benzene or petrol, allow the two liquids to separate, and run off the lower soap solution. Shake up the oily solution with distilled water once or twice and separate. Finally run off the solvent into a weighed flask or dish, evaporate off the solvent and weigh the residue, which will include unsaponifiable matter and the mineral oils in case of a mixture. Calculate percentage found.

Weight × 20 = unsaponifiable matter.

Epitome.

Saponify 5 grams, evaporate off alcohol.
Dissolve soap with water, residue with benzene.
Separate and weigh residue.

(74*c*) **Iodine Number.**—This is only carried out on vegetable and animal oils to ascertain presence of adulterants. Briefly, the process consists in treating a known quantity (about ·25 gram) of the oil dissolved in 10 c.c. of chloroform, or carbon tetrachloride, with a measured quantity of standard iodine solution (Hubl, Hanus, or Wijs). The excess of iodine is then titrated with standard thiosulphate solution, using starch as indicator. A blank experiment must be carried out at the same time. For works' purposes this estimation is seldom required.

(74*d*) Lubricating oils may also be tested for rosin and rosin oil, which will be also included in the saponifiable matter. Heat about 20 grams of the oil with 50 c.c. of alcohol (free from rosin) on the steam bath for a quarter of an hour. Decant the alcohol, evaporate to dryness, and add 5 c.c. of acetic anhydride, warm, cool, and add one drop of 18E H_2SO_4. Rosin or rosin oils give a fugitive violet colour. Cheap petroleum jellies and semi-solid lubricants should especially be tested for rosin.

(74*e*) Sulphur in light oils may be tested for, as follows:—Add a small piece of bright metallic sodium to about 100 c.c. of the oil in a flask, and boil cautiously under a reflux condenser for one hour. Cool, add water, drop by drop, until the sodium is dissolved, separate the aqueous liquid, and add a drop of freshly made sodium nitroprusside solution. A violet colouration indicates presence of sulphur.

WATER ANALYSIS.—The examination of a water on a works is usually undertaken to ascertain the suitability for use in boilers, etc., rather than to judge of its fitness for drinking and domestic purposes. As a matter of fact, a works laboratory is rarely suitably equipped or circumstanced to permit of such an analysis being conducted with any hope of obtaining reliable results. The processes necessary to judge of the suitability of water for drinking purposes are, therefore, only very briefly described.

(75) **Collection of Samples.**—Clear glass Winchester quart bottles are very suitable for collecting and storing water samples. Before being filled with the water to be tested, they should be thoroughly washed until free from all traces of acid, ammonia, etc., that they may have contained. Bottles which

oils in case of a mixture. Calculate percentage found.

Weight × 20 = unsaponifiable matter.

Epitome.

Saponify 5 grams, evaporate off alcohol.
Dissolve soap with water, residue with benzene.
Separate and weigh residue.

(74*c*) **Iodine Number.**—This is only carried out on vegetable and animal oils to ascertain presence of adulterants. Briefly, the process consists in treating a known quantity (about ·25 gram) of the oil dissolved in 10 c.c. of chloroform, or carbon tetrachloride, with a measured quantity of standard iodine solution (Hubl, Hanus, or Wijs). The excess of iodine is then titrated with standard thiosulphate solution, using starch as indicator. A blank experiment must be carried out at the same time. For works' purposes this estimation is seldom required.

(74*d*) Lubricating oils may also be tested for rosin and rosin oil, which will be also included in the saponifiable matter. Heat about 20 grams of the oil with 50 c.c. of alcohol (free from rosin) on the steam bath for a quarter of an hour. Decant the alcohol, evaporate to dryness, and add 5 c.c. of acetic anhydride, warm, cool, and add one drop of 18E H_2SO_4. Rosin or rosin oils

The sediment should be well searched for the lower forms of diatomaceæ and algæ, some of which cause a water to smell as if contaminated with dead fish.

Any great amount of suspended matter may be estimated by filtration of one or two litres of the water through a tared filter. The filter and contents are dried at 100° C. and weighed.

Increase in weight = suspended matter per litre.

The inorganic matter in suspension may be estimated by igniting the filter and contents in a crucible; recarbonate by adding a drop of ammonium carbonate, and ignite again at a low temperature and weigh.

Weight − ash of filter insoluble mineral matter per litre.

Epitome.

Filter 1,000 c.c. through tared paper and weigh.

Before proceeding with the chemical analysis it is well to ascertain whether to report as grains per gallon or parts per 100,000. Most engineers will probably prefer results in grains per gallon. It is really unimportant, if the report is to be used by intelligent men, as it is only necessary to multiply grains per gallon by ten and divide by seven or *vice versâ* with parts per 100,000 to convert one into the other.

(78) **Total Solids.** —Evaporate 250 c.c. of the water to dryness in a clean dry platinum or porcelain weighed evaporating basin, at first over an argand or bunsen burner, and finally on the water bath. Allow to cool in a good desiccator and weigh rapidly, as the solids are often very hygroscopic.

Weight − weight of dish × 400 = parts per 100,000.

Epitome.

Evaporate 250 c.c. to dryness and weigh.

(79) A qualitative examination may be made of the solids; if a quantitative analysis is required it will be necessary to evaporate 1,000 c.c. or more of the water to dryness. Estimate SiO_2, Al_2O_3, Fe_2O_3, P_2O_5, CaO, MgO, CO_2, SO_3, as in ordinary analysis. See paragraphs Nos. 5, 7, 8, 9, 11, 13, 14, 39. Salt will be found separately. Sodium and potassium may be estimated or reported by difference. The chief constituents are $CaCO_3$, $CaSO_4$, $MgCO_3$, $MgSO_4$, and organic matter in solution in the case of peaty waters.

(80) **Chlorine as Chlorides.**—A standard solution of silver nitrate containing 4·790 grams per litre will be required; also a dilute solution of potassium chromate free from chloride. Measure 100 c.c. of the water to be tested into a clean flask and add a drop or two of the chromate solution. Run in the standard silver nitrate solution until a

permanent orange tint is obtained. To ascertain the end point needs some little practice, and results should always be duplicated.

Read off from the burette the number of c.c. of silver nitrate used.

Number of c.c. parts per 100,000 of Cl.
Chlorine 1·65 parts per 100,000 of NaCl.

Epitome.

Titrate 100 c.c. with standard silver nitrate, using potassium chromate as indicator.

(81) **Hardness.**—The hardness of a water is a factor of great importance on a works. Hardness is divided into two classes: "permanent," mainly due to sulphates of calcium and magnesium (and the other alkaline earths); and "temporary," due to the carbonates of these metals, the latter being removable by boiling.

(82) *Estimation of hardness by standard soap solution.*—The following standard solutions are required. Standard hard water made by dissolving without loss 0·2 gram of powdered calcite ($CaCO_3$) in dilute hydrochloric acid and evaporating to dryness on the water bath several times until quite free from acid. The $CaCl_2$ is then dissolved and made up to 1,000 c.c. with pure distilled water.

One c.c. = ·0002 gram of $CaCO_3$ or 20 parts per 100,000.

(83) *Standard Soap Solution.*—A standard potassium oleate soap solution may be readily prepared by dissolving 80 grams of pure oleic acid in "proof" spirit and exactly neutralising with alcoholic potassium hydrate solution, using a drop or two of phenolphthalein as indicator. The solution is then titrated with the standard hard water (82).

(84) *Standardisation of the Soap Solution.*—Measure 100 c.c. of standard hard water into a clean glass-stoppered bottle, and run the soap solution into the bottle a few c.c. at a time until a lather begins to form; then add the soap solution more cautiously, inserting the stopper and shaking between each addition. When the reaction is nearing completion the contents of the bottle will only give a faint dull sound. Soap solution must be added until a permanent lather, which persists for at least two minutes, is obtained. The soap solution must be then so diluted that the 100 c.c. of water require exactly 21 c.c. to produce a lather, the extra c.c. being required to produce a lather with 100 c.c. of distilled water.

Suppose the water only requires 16 c.c. of soap, then every 16 c.c. of the solution must be diluted with a mixture of about 2 to 1 alcohol and water to make 21 c.c. Rectified alcohol or industrial methylated spirit must be used, as ordinary methylated spirit produces a ppt. with water.

One c.c. of the standard soap solution will thus = 1 part of $CaCO_3$ per 100,000. If 70 c.c. of water is taken, results as expressed in degrees on Clarke's scale will be obtained—*i.e.*, 1° = 1 grain of $CaCO_3$ per gallon.

(85) *To ascertain temporary and permanent hardness of a water.*—Measure 100 c.c. of the water under examination into the titration bottle; or, if the water is known to be very hard, take 50 c.c. and dilute with distilled water. Run in the soap solution cautiously as described until a permanent lather is obtained; deduct 1 c.c. from the amount used: then number of c.c. = degrees of total hardness.

Epitome.

Titrate 100 c.c. with standard soap.

(86) *Permanent hardness.*—Boil, in a flask or beaker, 100 c.c. of the water down to a volume of about 50 c.c. or rather less. Allow to cool, filter or decant, and make up to 100 c.c. again with distilled water. Titrate with soap solution as before.

Number of c.c. = parts per 100,000 of permanent hardness.

Total – permanent = temporary hardness.

Epitome.

Titrate 100 c.c., after boiling, with standard soap.

(87) *Estimation of hardness by Standard Acid—temporary.*—To 500 c.c. of the water, or less if

very hard, tinted with methyl orange, run in from a burette $\frac{N}{10}$ H_2SO_4 until a red colouration is just produced.

Number of c.c. used = parts per 100,000 of $CaCO_3$ as "temporary hardness."

(88) *Permanent hardness.*—To 250 c.c. of the water add 50 c.c. of $\frac{N}{10}$ Na_2CO_3 solution and boil for about half an hour, or, if magnesium salts be present, evaporate to dryness.

Filter off the ppt., or extract the residue with boiled distilled water, filter and wash; when cool, make the filtrate up to 250 c.c. with distilled water.

Titrate 50 c.c. of the filtrate with $\frac{N}{10}$ H_2SO_4, using methyl orange as indicator.

Since 10 c.c. of $\frac{N}{10}$ $NaCO_3$ was present in every 50 c.c. of water, then 10 − number of c.c. of acid used = number of c.c. of soda removed. Call this x; then soda used by the 250 c.c. of water = $(x \times 5)$; as 1 c.c. of $\frac{N}{10}$ Na_2CO_3 = ·005 gram of $CaCO_3$, then $(x \times 5) \times \cdot005 \times 400$ = parts of $CaCO_3$ per 100,000 present as permanent hardness.

(89) *Estimation of hardness in a softened water or water containing Soda.*—In a water that gives

an alkaline reaction to litmus or methyl orange there can be no premanent hardness, as carbonates of calcium and magnesium will be pptd. on boiling. Boil 250 c.c. or 500 c.c. of the water, filter off ppt., if any, wash and make up filtrate to original bulk, titrate whole or aliquot part of the solution with $\frac{N}{10}$ H_2SO_4, and calculate to Na_2CO_3 parts per 100,000.

Number of c.c. of acid used × 200 (or 400) × ·0053 = parts per 100,000 Na_2CO_3.

Contamination.—To ascertain whether water is contaminated with sewage or with the products of animal or vegetable decomposition, it is necessary to estimate the free or saline ammonia, and the albuminoid ammonia which is derived from nitrogenous matter, by boiling with alkaline permanganate of potassium.

The following special reagents are required For full particulars of the process involved a work on water analysis should be consulted.

The estimation must be conducted under conditions that admit of no contamination from fumes of ammonia, etc. The apparatus and distilled water used must also be quite free from any traces of ammonia or ammonium salts.

(90) *Standard Ammonium Chloride.*—Dissolve 3·147 grams of pure ammonium chloride in

ammonia-free distilled water, make up to 1 litre and label as "stock NH_4Cl solution."

Take 10 c.c. of (90) and dilute to 1,000 c.c.

1 c.c. = ·00001 gram of NH_3.

(91) *Nessler's Reagent.*—Dissolve 35 grams of potassium iodide in about 250 c.c. of distilled water. Then add gradually a cold saturated solution of mercuric chloride, stirring constantly until a faint permanent ppt. is formed; allow to stand and decant off. Then add when cool a solution made by dissolving 150 grams of caustic potash in 150 c.c. of water. Add a drop or two of mercuric chloride solution until a slight ppt. is formed. Dilute to 1 litre and allow to stand. Decant off a portion into a smaller bottle for use, and always use a pipette or graduated tube to measure out the required quantity of solution.

(92) *Alkaline Permanganate.*—Dissolve 200 grams of caustic potash in about 800 c.c. of ammonia-free distilled water. Then add 8 grams of potassium permanganate, allow this to dissolve and boil for a short time; when cool, make up to 1 litre.

(93) *Estimation of "Free Ammonia."*—Distil 250 c.c. of the water, or less if very contaminated, using a large flask fitted to a Liebig's condenser in the usual way. The apparatus must first be well cleaned and boiled out with ammonia-free water until the distillate gives no reaction with Nessler's reagent. The distillate from the water under

examination is collected in a series of Nessler tubes, and the operation continued as long as a colouration is produced with 2 c.c. of the reagent (91).

Usually three cylinders are sufficient. To each add 2 c.c. of the reagent and prepare a similar tube using distilled water; measure into it 1 c.c., or more, of the standard ammonium chloride and then add 2 c.c. of Nessler reagent. Tubes are thus prepared to match the tint of each tube of distillate, and the total number of c.c. of standard ammonium chloride required noted.

Number of c.c. used × 0·0001 × 400 = parts of free ammonia per 100,000.

The Nessler must always be added to the ammonium chloride and not *vice versâ*. Instead of a flask and Liebig's condenser a large retort fitted to a spherical condenser placed in a bath of water may be used. Quite 100 c.c. of distillate is collected and tested against standard ammonium chloride as described.

Epitome.

Distil 250 c.c. of water.
Test distillate with Nessler reagent (91).

(94) *Albuminoid Ammonia.*—To the water remaining in flask or retort add 25 c.c. of the alkaline permanganate (92), and continue the distillation as long as possible. Then test the distillate as before, and ascertain number of c.c. of ammonium

chloride solution required to match depth of tint. The calculation is the same as above.

Epitome.

Add 25 c.c. of alkaline permanganate and redistil.

For interpretation of results consult a work on water analysis. See *Appendix* 33A for typical analyses.

(95) *Estimation of Oxygen required to oxidise Organic Matter.*—A standard solution of potassium permanganate is required containing 0·395 gram per litre. Then 1 c.c. ·0001 gram of oxygen.

Into a clean glass bottle or flask place 250 c.c. of water to be tested; then add, from a graduated pipette or tube, 1 c.c. of the standard permanganate solution and about 10 c.c. of sulphuric acid to which has been added enough permanganate to make it slightly pink. Allow to stand for fifteen minutes at 15° C. If the pink colouration remains, the oxygen absorbed is nil; if it disappears, add another c.c. and continue until the colouration is permanent for one hour.

If the water is likely to use a lot of permanganate solution, take 250 c.c. in flask as before, and 250 c.c. of distilled water in another flask; add 10 c.c. of permanganate and 10 c.c. of the dilute acid to each, and allow to stand for three hours at the same temperature (15° C.). Then add a drop or two of potassium iodide solution to the distilled water as "check," and titrate with a solution of sodium thiosulphate containing 1 gram per 1,000 c.c., using freshly made starch solution as indicator. Note the number of c.c. of thiosulphate required to just remove the blue colour caused by the iodide and starch. Repeat the titration in a similar way with the water under examination.

Note number of c.c. of thiosulphate used. As the distilled water uses up no $K_2Mn_2O_8$, the equivalent in terms of thiosulphate will be known.

(96) *Estimation of Nitrates + Nitrites.*—The total solids from 500 c.c. of water are taken up with a small quantity of distilled water, filtered, and washed. The filtrate, after being evaporated to about 2 c.c., is put into the cup of a nitrometer and then carefully into the instrument, which must be full of clean mercury. The beaker and cup are then washed with a little water, which is also allowed to run into the graduated tube; 6 c.c. of 36E H_2SO_4 free from nitrates are then carefully admitted, care being taken not to admit air. Any air or CO_2 bubbles which may be produced are carefully driven out by raising the other limb of the instrument. The tap is again turned off, and the tube and contents carefully but thoroughly shaken for about ten minutes, or until there is no further increase in the volume of gas collected. Allow to cool, and then adjust the level of the mercury in both tubes until that in

the open tube is about one-tenth of the volume of the liquid above the mercury in the closed tube. Read off the volume of nitric oxide (NO) and note temperature and barometric pressure.

Calculate to volume at 0°, and 760 mm. NO contains half its volume of nitrogen, so the gas thus found represents nitrogen in 1,000 parts. 1 c.c. of nitrogen weighs ·0012544 gram, therefore:

c.c. of N (at NTP) × ·12544 = parts of N per 100,000.

$$N \times 4{\cdot}5 = HNO_3.$$

(97) *Estimation of Nitrates using Standard Indigo Solution.*—The following standard solutions are required:

Indigo Solution.—Digest 1 gram of pure solid indigo with 10 c.c. of fuming sulphuric acid for several hours on a water bath until all is dissolved. Make the solution up to a volume of two litres with distilled water.

Standard Nitrate.—Dissolve 1·604 grams of pure KNO_3 in one litre of distilled water. This contains the equivalent of 1 gram per litre of HNO_3: for use dilute ten times; then

$$1{,}000 \text{ c.c.} = 0{\cdot}1 \text{ gram } HNO_3.$$

To standardise Indigo.—Take 10 c.c. of the dilute nitrate solution and 10 c.c. of distilled water in a flask, add 20 c.c. of pure 36E H_2SO_4 free from nitrates, and immediately run in from a previously filled burette the indigo solution, a few drops at a time, until the first brown tint begins to become darker, when the addition may be hastened. The reaction is complete when the solution assumes a decidedly green tint, which is best seen upon dilution. With a little practice the end tint is soon recognised.

$$\frac{\cdot 001}{\text{No. of c.c. used}} = \text{strength of indigo per c.c.}$$

To estimate Nitrate in Water.—Take 20 c.c. of the water and 20 c.c. of 36E H_2SO_4 and titrate as described. If the water contains more than 10 parts of HNO_3 per 100,000, use 10 c.c. and dilute to 20 c.c. with distilled water.

No. of c.c. used × factor × 50,000 = parts per 100,000 of HNO_3.

The indigo should occasionally be checked, as it deteriorates after a time. An estimation can be carried out in less than five minutes.

(98) *Poisonous Metals.*—A drinking water should always be tested for lead, copper, etc., by passing H_2S gas through a quantity of the water contained in a clear glass vessel. No dark colour should be produced. If present, lead may be estimated by evaporating several litres of the water to a small bulk, acidifying slightly with hydrochloric acid and ppt. with H_2S.

Filter, wash, convert into $PbSO_4$, and weigh; or lead may be estimated colorimetrically by comparison of the depth of tint produced by adding

H_2S to a known amount of water, and also to a standard dilute solution of lead nitrate.

Epitome.

Pass H_2S through at least 1 litre of water.
Estimate as $PbSO_4$ or colorimetrically.

Water softening.—Many systems of water softening are in vogue; they are nearly all based upon the removal of carbon dioxide and consequent precipitation of calcium carbonate by means of lime, or precipitation of carbonate and sulphates by means of soda ash (Na_2CO_3) or caustic soda NaOH.

The following are the principal reactions involved:

(1) $CaCO_3 + CO_2 + Ca(OH)_2 = 2CaCO_3 + H_2O$

(2) $MgCO_3 + CO_2 + 2Ca(OH)_2 = 2CaCO_3 + Mg(OH)_2 + H_2O$

(3) $CaSO_4 + Na_2CO_3 = CaCO_3 + Na_2SO_4$

(4) $MgSO_4 + Ca(OH)_2 + Na_2CO_3 = CaCO_3 + Mg(OH)_2 + Na_2SO_4$

(5) $CaCO_3 + 2NaOH = Ca(OH)_2 + Na_2CO_3$

(6) $CaSO_4 + 2NaOH = Ca(OH)_2 + Na_2SO_4$

The following illustrates the method of calculating the amount of lime required per 1,000 gallons, the $CaCO_3$ present being known from the temporary hardness or by analysis of the total solids:

$$\begin{array}{lllll} CaCO_3 & + CO_2 & + CaO & = & 2CaCO_3 \\ 100 & & + 56 & & \end{array}$$

100 parts of $CaCO_3$ requires 56 parts of quicklime (CaO).

The water has say 15 parts of $CaCO_3$ per 100,000.

1,000 parts of water contain ·15 of $CaCO_3$, therefore 1,000 parts of water require $\frac{56 \times \cdot 15}{100}$;

$$\therefore \frac{56 \times \cdot 15 \times 70}{100} = \text{grains of } CaO \text{ per gallon.}$$

$$\frac{56 \times \cdot 15 \times 70 \times 1{,}000}{100} = \text{grains of } CaO \text{ per } 1{,}000 \text{ galls.,}$$

or $56 \times \cdot 15 \times 700 = 5{,}880$ grains;

there are 7,000 grains per lb. (16 oz.),

$$\frac{5{,}880 \times 16}{7{,}000} = \text{oz. per } 1{,}000 \text{ gallons;}$$

or, as one equation, $\frac{56 \times \text{degrees of hardness} \times 16}{1{,}000}$.

In actual calculations the percentage of CaO in the lime used must be known (140) and allowed for. Slaked lime is always used in practice: the equation then becomes

$$\frac{74 \times \text{degrees of hardness} \times 16}{1{,}000} = \left\{ \begin{array}{l} \text{oz. of } Ca(OH)_2 \text{ per} \\ 1{,}000 \text{ gallons.} \end{array} \right.$$

And $\frac{74 \times \text{hardness} \times 16 \times 100}{1{,}000 \times \text{per cent. of } Ca(OH)_2 \text{ in lime}} =$ oz. per 1,000 gallons of slaked lime required.

As the amount of CO_2 in the water is always largely in excess of that required to keep the $CaCO_3$ in solution, the above calculation will only give approximately the amount of lime to be actually used.

The following method of ascertaining the amount of lime (CaO) required per million parts of an ordinary water is taken from a paper by Mr. W. D. Collins in the *Engineering Record*, February 16th, 1907 (U.S.A.):

(99) *Excess of* CO_2.—Titrate 100 c.c. of the water with $\frac{N}{50}$ Na_2CO_3 solution free from bicarbonate, using phenolphthalein as indicator.

No. of c.c. used × 10 = $CaCO_3$ equivalent of CO_2 (*a*).

(100) *Temporary hardness.*—Titrate 100 c.c. of the water with $\frac{N}{50}$ H_2SO_4 in a 200-c.c. graduated flask, using methyl orange as indicator.

No. of c.c. used × 10 = parts per million of $CaCO_3$ (*b*).

(101) *Magnesia.*—Heat to boiling in the 200-c.c. flask the neutralised water (100); boil for fifteen minutes, add 25 c.c. of saturated lime water. Make up to 200 c.c. at temperature of room. Filter into a graduated cylinder, reject first 50 c.c., titrate next 100 c.c. with $\frac{N}{50}$ H_2SO_4, using methyl orange as indicator.

Repeat, using distilled water.

Difference = amount of H_2SO_4 which has been neutralised by $Ca(OH)_2$ required to ppt. MgO.

This No. of c.c. × 20 = parts per million of $CaCO_3$ equivalent to magnesia (*c*).

(102) *Permanent hardness.*—Boil 250 c.c. of the water in a porcelain dish. Add 25 c.c. of $\frac{N}{10}$ soda reagent (equal parts Na_2CO_3, NaOH), and boil for ten minutes. Filter, make up to 250 c.c., and titrate 100 c.c. with $\frac{N}{50}$ H_2SO_4. Repeat, using distilled water. Difference in number of c.c. of H_2SO_4 required = soda reagent used.

This number × 10 = parts per million of $CaCO_3$ as permanent hardness (*d*).

(103) Then $a+b+c \times \cdot 56$ = parts per million of CaO required to soften water.

$d \times 1{\cdot}06$ = parts per million of soda required to remove permanent hardness.

GAS ANALYSIS.—Collection of Samples—A sample of rotary kiln or furnace gas can best be obtained at the base of the shaft. For this purpose a brass or copper tube sufficiently long is introduced through an eye hole or an opening especially made.

The length of tube inserted in the furnace should have holes pierced in the sides in order to obtain a sample of the gas from as many different points as possible.

In order to prevent corrosion by acid gases, the tube may be dipped in a strong solution of borax and dried; upon becoming heated the borax fuses and forms a protective glaze. Aspirators of metal or glass may be purchased, but for ordinary use a very effective one can be easily constructed from a Winchester quart bottle as follows:

(104) Fit the bottle with a sound cork or rubber bung pierced with two holes. The cork is fitted with two glass tubes bent at right angles, one being sufficiently long to reach to the bottom of the bottle, the other only just passing through the cork.

When the apparatus is to be used a glass or rubber tube sufficiently long to reach below the level of the bottom of the bottle is attached to the exterior end of the longer tube, the bottle is filled with water and suction applied to this tube; the water is thus syphoned off, and the gas enters the other tube and fills the bottle. The rate of aspiration may be regulated by placing a screw clamp on the rubber connection. By preparing a number of bottles fitted in a similar way, but the glass tubes bearing glass stop-cocks, any number of samples of gas may be obtained with the use of one aspirator.

(105) The apparatus is fitted up as follows:—Place the sampling tube in the flue; to the end obtruding attach a wash bottle or a dust trap consisting of a tube loosely packed with asbestos or cotton wool; connect this to the sample bottle and

that in turn to the aspirator, making sure beforehand that all connections and stoppers are absolutely gas tight. Before sealing off and disconnecting the sample bottle, aspirate at least twice its volume of gas through the apparatus in order to remove air and to saturate the wash water with the gas under examination.

Analysis of Gases in the Hempel Apparatus.—In kiln or furnace gas it is necessary to estimate the following constituents when present: Carbon dioxide, carbon monoxide, oxygen, nitrogen, and hydrocarbons. For a description of the apparatus, mode of fitting together, and other details Hempel's *Gas Analysis* should be consulted.

The following solutions for absorption are required:—

(106) *For Carbon Dioxide.*—Dissolve 160 grams of KOH in 130 c.c. of distilled water.

(107) *For Oxygen.*—Dissolve 10 grams of pyrogallol in 200 c.c. of potash solution (106).

(108) *For Carbon Monoxide.*—Dissolve a mixture of 86 grams of copper oxide (CuO) and 17 grams of copper filings in 1,086 grams (969 c.c.) of hydrochloric acid of sp. gr. 1·124, adding this mixture to the acid slowly with frequent stirring. Store in a bottle containing metallic copper.

(109) *Filling the Burette.*—Connect a piece of

capillary glass tube to the top of the measuring tube of the burette by means of a piece of stout rubber tubing, which should carry a clip and be wired on. Fill the pressure tube with water saturated, if possible, with the gas under examination; raise the tube, thus causing the water to enter the measuring tube until it issues from the capillary tube. Close the clip and connect to the aspirator by means of another piece of short rubber tube. All rubber connections should be wired on. Lower the pressure tube and open the clips until the gas to be analysed is drawn in and rather more than 100 c.c. are contained in the measuring tube.

Close the clips and disconnect from the aspirator, which must, of course, be again securely sealed up. Now close the long rubber tube between the pressure and measuring tubes. Raise the pressure tube and open the clip to allow water to flow into the measuring tube until it just reaches the graduation. Again close the clip on the connecting tube and open the top of the measuring tube; excess of gas will escape, and exactly 100 c.c. will remain at atmospheric pressure; close the clips.

For technical work it is not necessary to note barometric pressure or temperature, provided they do not alter during the examination.

(110) *Estimation of Carbon Dioxide.*—Fill an ordinary absorption burette with the potash solution (106) by means of a funnel attached to the tube connected with the reservoir bulb. Then, by

means of suction, draw the solution into the other (absorption) bulb until it almost completely fills the capillary tube; thus practically all air is excluded from the apparatus. Such an amount of reagent must be used that it will be contained by the reservoir bulb when it is driven back by the gas from the pipette. Having filled the burette, note the position of the reagent in the capillary tube and bring it to the same position between each reading. Attach the pipette by means of the connecting capillary tube and rubber joint.

Open the pinch-cocks and raise the pressure tube, thus causing the gas to enter the burette Allow the water to fill the connecting capillary tube, close the pinch-cocks, and gently shake the contents of the burette in order to present a larger surface of the reagent to the gas. Allow to stand for at least five minutes. Cause the gas to return to the pipette by lowering the pressure tube adjust the level of the water, allow to stand for a few minutes, and then read off the volume of the gas. Repeat the operation until a constant volume is obtained.

100 − reading = volume of carbon dioxide per cent.

Epitome.

Treat 100 c.c. with potash solution in gas pipette.

(111) *Estimation of Oxygen.*—The pipette is

replaced by a "double" one containing alkaline pyro-gallate solution (107), and the absorption process repeated.

Diminution in volume = oxygen per cent.

(112) *Estimation of Carbon Monoxide.*—The pipette is replaced by one containing acid cuprous chloride (108), and the absorption process repeated.

Diminution in volume = carbon monoxide per cent.

(113) *Nitrogen.*—The residual gas is generally taken as consisting of nitrogen, and this is the case when the furnace is working properly. Should there be an insufficient supply of air, there may be hydrocarbons or hydrogen present.

(114) *Estimation of Combustible Gases.*—About 30 c.c. of the residual gas are made up to 75 c.c. with oxygen, and the exact volume noted. The mixture is then fired in an explosion pipette or eudiometer by means of an electric spark. The contraction in volume is noted and the gas passed into the pipette containing potash solution (106), which absorbs the CO_2 formed, and the volume again noted.

The volume of hydrogen and methane may be calculated as follows:

Let x = contraction due to firing.
y = „ after absorption of CO_2.
Vol. of hydrogen equals $\frac{2}{3}(x - 2y)$
„ methane equals y.

The percentage is then calculated as follows:

$$\left.\begin{array}{l}\text{Vol. of hydrogen}\\ \text{or methane}\end{array}\right\} \times \frac{\text{Vol. of residual gas}}{\text{Vol. taken for combustion}} \times 100.$$

(115) *Analysis with the Orsat Apparatus.*—This is a portable apparatus consisting of a measuring burette and absorption pipettes, which are filled with the usual reagents. Some forms of the apparatus also have a palladium tube for the determination of the combustible gases. According to Hempel, there are several sources of inaccuracy.

Full instructions are usually supplied with the instrument. It is a convenient apparatus for control work.

CHAPTER V.

CEMENT ANALYSIS.

(116) In order to ascertain whether the chemical composition of a sample of cement fulfils the requirements of the British standard specification, it is necessary to estimate silica, insoluble matter, alumina, magnesia, sulphuric anhydride, and loss on ignition. It is not actually necessary to estimate iron oxide, but it somewhat simplifies the analysis to do so, in which case the alumina can, in the absence of phosphates, be estimated by difference.

For particulars of the requirements the standard specification should be consulted. So far as the lime, silica, and alumina ratio is concerned, the following formula may be employed :—

Let x = per cent. CaO, y = per cent. SiO_2, and z = per cent. Al_2O_3, then

$$\frac{\frac{x}{56}}{\frac{y}{60}+\frac{z}{120}}$$

should fall between 2·85 (maximum) or 2·0 (minimum).

(117) **Silica + Insoluble.**—Weigh out into a 6-in. flat porcelain or platinum dish 0·5 gram of the cement. Add a little distilled water, and rotate the dish to prevent setting; then add 25 c.c. of pure 10E HCl, place on a sand or water bath, and evaporate to dryness. Cover with a clock-glass, and bake on a hot plate at a temperature of 200° C. for at least one hour. Remove from hot plate, and allow to cool. Add 25 c.c. of 10E HCl and about the same amount of water, warm for a few minutes on the sand or water bath until the insoluble matter is quite free from iron compounds. Filter through a 12·5-cm. rapid filter paper, wash by decantation, remove every trace of silica adhering to the dish by means of a rubber-tipped glass rod. Wash with hot water until quite free from chloride. Return the filtrate to the dish, and again evaporate to dryness on the sand bath.

Take up with 25 c.c. 10E HCl and water, digest if necessary, filter off any trace of silica through a 9-cm. paper, and wash well. Dry and ignite the two residues together for at least one hour in a good muffle furnace.

Weight × 200 = silica + insoluble.

Treat with hydrofluoric acid in platinum crucible as under (17).

(118) **Alumina and Ferric Oxide.**—Return the filtrate from (117) to the evaporating dish, add a drop or two of bromine water, and bring nearly to

the boiling point over an argand burner or on a hot plate. Cautiously add a slight excess of 10E NH_4OH, and gently heat until nearly all the excess of ammonia has been driven off. Filter through a 15-cm. rapid paper; wash slightly, and collect the filtrate in a large Phillip's beaker. Pierce a hole in the filter paper and wash the ppt. back into the original dish. Without removing paper from the funnel, drop on to it 15 c.c. of 10E HCl, and wash down into the dish with about 20 c.c. of hot water. Reprecipitate the alumina and iron oxide as before, filter, and wash thoroughly and rapidly, well churning up the ppt. Dry, and ignite in muffle for one hour, cool in desiccator and weigh.

Weight × 200 = per cent. of Al_2O_3 + Fe_2O_3.

Fuse with sodium bisulphite and estimate iron volumetrically as in (17*b*), or take a fresh portion of cement (0·5 gram) and estimate iron volumetrically after separating silica as under (26) or (27).

(118*b*) *Separation of Alumina and Ferric Oxide.* —Precipitate the mixed hydroxides with ammonia as in (118), filter and wash.

Dissolve the ppt. on the filter with 10E HCl, and allow the solution to run directly into boiling 5E KOH, preferably contained in a platinum or nickel dish.

Filter off and wash the ppt. of ferric hydroxide;

this may be dissolved in 10E HCl and repptd. with 18E NH_4OH, filtered, washed, dried, ignited, and weighed as Fe_2O_3.

$$\text{Weight} \times 200 = \text{per cent. } Fe_2O_3.$$

Acidify the filtrate with 10E HCl, and ppt. the alumina with 10E NH_4OH, filter and wash.

Redissolve with 10E HCl and reppt. with 10E NH_4OH, filter, wash, dry, ignite, and weigh.

$$\text{Weight} \times 200 = \text{per cent. } Al_2O_3.$$

(119) **Lime** (CaO).—Evaporate the filtrate (118) somewhat, if necessary, and heat nearly to boiling, add about 20 c.c. of 5E NH_4OH, boil and add at least 30 c.c. of boiling $\frac{3E}{5}$ ammonium oxalate solution, and continue to boil for five minutes. Allow to stand in a warm place, if possible, for at least one hour. Filter through a 15-cm. paper and wash. The filtrate and first washings are reserved for MgO determination (121).

Continue the washings until a drop of the filtrate does not discolour an acidified very dilute solution of potassium permanganate. Estimate CaO gravimetrically (see *8a*, *8b*, and 9) or by means of standard solution of permanganate, as described in (10).

(120) For very accurate work ignite the still moist ppt. in a platinum crucible over a small bunsen burner. Place in a large beaker, dissolve ppt. in redistilled 10E HCl, dilute with 150 c.c.

of water, warm, add slight excess of 10E NH_4OH, filter off any traces of alumina, reprecipitate with ammonium oxalate, and estimate the CaO as before, or ignite in good muffle to constant weight.

Weight of CaO × 200 per cent. CaO.

(121) **Magnesia** (MgO).—The filtrate or filtrates from (119) are evaporated nearly to dryness in the large dish. Add 30 c.c. of pure 16E HNO_3, evaporate to dryness, and continue heating on the hot plate until all ammonium salts are volatilised. Remove from plate and dissolve residue in 5 c.c. of 10E HCl and about 20 c.c. of distilled water. Add one drop of bromine water and slight excess of 10E NH_4OH, warm carefully for a few minutes and then filter off any ppt. through a 5-cm. rapid paper. If not exceeding 0·002 gram, ignore. Collect filtrate in a 200-c.c. beaker, add 10 c.c. of 20E NH_4OH and 5 c.c. of $\frac{2E}{3}$ Na_2HPO_4 solution. Stir well with a rubber-tipped rod, and allow to stand in a cool place over night.

Filter through a 7 or 9 cm. filter paper, wash by decantation, using 5E NH_4OH ; then transfer ppt. to the paper, remove by means of the rubber-tipped glass rod any particles adhering to the sides of the beaker, and continue to wash until free from chlorides. The ppt. may be ignited while moist in a platinum or porcelain crucible in the muffle furnace or over a good burner for half an hour. Cool in a desiccator and weigh as $Mg_2P_2O_7$.

Weight of ppt. × 0·362 × 200 = per cent. of MgO (see *Appendix*, 25A). The ppt. before washing may be dissolved in a little warm water and a drop of hydrochloric acid, and repptd. by addition of 1 c.c. of $\frac{2E}{3}$ Na_2HPO_4 and excess of ammonia.

(122) **Alkalies.** These are generally estimated by difference; they may be estimated directly by the Lawrence Smith method as described under Clay (31), or as follows:—

Treat one gram of cement in a large platinum dish, as for ordinary analysis for estimation of SiO_2, Al_2O_3, Fe_2O_3, CaO. Evaporate filtrate from CaO determination to dryness in a platinum dish carefully. Ignite residue until free from ammoniacal salts, cool, then add 5 c.c. of water and brush in about 1 gram of powdered oxalic acid crystals. Evaporate carefully to dryness and ignite as before. Treat the residue with about 20 c.c. of hot distilled water, filter, and wash; residue consists of MgO and may be weighed as such.

The filtrate is made acid with 10E HCl and evaporated to dryness very carefully in a weighed platinum dish. Cool in a desiccator and weigh.

Weight = NaCl + KCl.

Na_2O and K_2O can then be estimated as under 32*b*.

Perform a blank experiment, using same quantities of reagents and deduct result from above.

Epitome.

Separate SiO_2, Al_2O_3, Fe_2O_3, CaO.
Evaporate to dryness, add oxalic acid and water, evaporate to dryness, take up with water, filter and wash. Acidify filtrate with hydrochloric acid and evaporate.
Separate KCl and NaCl by means of platinum chloride (see 32*b*).
For loss on ignition, see (130).

(123) **Ordinary Method of Analysis for Technical Purposes.**—*SiO_2 and insoluble.*—Weigh out 0·5 gram of cement into a 6-inch porcelain dish, add a little water, swirl round, and add 25 c.c. 10E HCl. Evaporate to dryness on hot plate, cover, and bake for one hour. Remove from hot plate, and when nearly cool take up with 25 c.c. 10E HCl and water. Digest, if necessary, filter off SiO_2 + insoluble, clean dish, well wash, ignite and weigh.

Weight × 200 = per cent. silica and insoluble.

(124) *Al_2O_3, Fe_2O_3.*—To filtrate from (123) contained in the large dish add a drop or two of bromine water and slight excess of 10E NH_4OH; gently boil off excess of ammonia, filter through a rapid paper, wash well, ignite and weigh.

Weight × 200 = per cent. Al_2O_3, Fe_2O_3.

Estimate Fe_2O_3 separately if required (26) or (27).

(125) *CaO.*—Boil filtrate from (124) in a large Phillips beaker, add slight excess of ammonia and 50 c.c. of boiling $\frac{3E}{5}$ ammonium oxalate, boil for three minutes, allow to stand for half an hour, filter, wash well, and estimate by means of standard permanganate (9).

(126) Evaporate filtrate from (125) nearly to dryness, add 30 c.c. of 16E HNO_3 and drive off ammonium compounds. Take up with a few drops of hydrochloric acid and a little water. Add slight excess of ammonia, filter off any ppt., to filtrate add 10 c.c. 10E NH_4OH and 5 c.c. of $\frac{2E}{3}$ Na_2HPO_4, stir well or shake in a stoppered bottle vigorously, allow to settle, filter, wash with 5E NH_4OH, and weigh as $Mg_2P_2O_7$ (25A).

Weight × 0·362 × 200 = per cent. MgO.

Epitome.

0·5 gram cement, treat with 25 c.c. hydrochloric acid, evaporate to dryness. Bake one hour, allow to cool, take up with 25 c.c. hydrochloric acid. Filter, wash, ignite, and weigh = SiO_2 + insoluble. Ppt. Al_2O_3, Fe_2O_3 with ammonia. Filter, wash, ignite, and weigh = Al_2O_3, Fe_2O_3. To filtrate add boiling ammonium oxalate + ammonia. Filter, wash, estimate = CaO.

Evaporate filtrate nearly to dryness, add 30 c.c. nitric acid, drive off all Am. salts. Dissolve in dilute hydrochloric acid, ppt. and filter off traces of Al_2O_3, Fe_2O_3; ignore ppt. To filtrate add ammonia + sodium phosphate, agitate, allow to settle, filter, wash with 5E NH_4OH, ignite and weigh as $Mg_2P_2O_7$; calculate to MgO.

(127) **Insoluble Residue.**—Weigh out into a 5-in. flat distilled dish 0·5 gram of cement. Add a little distilled water and rotate to prevent setting; then add 10 c.c. of 10E HCl and warm on the hot plate for ten minutes or a quarter of an hour.

Add 10 c.c. of 10E HCl and about 25 c.c. of water, allow insoluble matter to settle, filter through a 9-cm. rapid paper, wash at least three times by decantation, allowing as little of the insoluble matter as possible to leave the dish. Finally, wash back into the dish any particles adhering to the filter paper without removing latter from the funnel. The filtrate is used for estimation of SO_3 (128). To the contents of the dish add 10 c.c. of 3E Na_2CO_3 solution and boil for ten minutes. Filter rapidly through the paper previously used, and wash with boiling water until a drop of the filtrate leaves no residue upon evaporation. Dry, ignite, and weigh.

Weight × 200 = per cent. insoluble residue.

The weight so obtained is subtracted from the total SiO_2 and insoluble residue (117) and (123).

Epitome.

Treat 0·5 gram with warm hydrochloric acid.
Filter, wash by decantation.
Boil residue with 10 c.c. sodium carbonate solution.
Filter, wash, ignite, and weigh.

(128) **Sulphuric Anhydride** (SO_3).—Boil in a 10-oz. Phillips beaker the filtrate from (127), and whilst still boiling add drop by drop 10 c.c. of E $BaCl_2$ solution; after five minutes allow to settle in a warm place for at least two hours, if possible. Filter through a 7-cm. close-texture paper, wash with warm water until quite free from chloride. Dry, ignite, and weigh as $BaSO_4$. The crucible containing the damp paper may be placed in the mouth of the muffle furnace and ignited therein afterwards without much fear of an inaccurate result.

Weight of $BaSO_4 \times 0·3431 \times 200 =$ per cent. SO_3.

Epitome.

Precipitate with barium chloride solution.
Wash, ignite, and weigh as $BaSO_4$.

(129) **Sulphur as Sulphide.**—Sulphides, if present in sufficient amount, may be estimated by the following method:—Treat 0·5 or 1 gram of cement with a little water in the usual way, then add 25 c.c. of 16E HNO_3, warm and evaporate gently to dryness, allow to cool, and take up with

10 c.c. of 10E HCl and water. Filter off silica and wash.

To the filtrate add 10 c.c. of E $BaCl_2$ solution and treat as in (128). From the weight of $BaSO_4$ obtained deduct the equivalent found when estimating SO_3. The excess $BaSO_4 \times 0{\cdot}137 \times 200$ (or 100) = per cent. sulphur as sulphide.

Epitome.

Treat with nitric acid, evaporate, take up with hydrochloric acid, filter and wash.

To filtrate add $BaCl_2$ solution, filter; wash, weigh as $BaSO_4$.

The sulphur found as sulphide may be calculated to calcium sulphide, CaS, thus:

$$\text{Weight of } BaSO_4 \times 0{\cdot}309 \times 200 = CaS,$$

in which case the equivalent must be deducted from the lime (CaO) found. One per cent. of CaS = 0·776 per cent. CaO. In like manner SO_3 may be converted into $CaSO_4$.

$$\text{Weight of } BaSO_4 \times 0{\cdot}583 \times 200 = CaSO_4.$$

For each 1 per cent. of $CaSO_4$ deduct 0·41 per cent. from the CaO found.

(130) **Loss on Ignition.**—0·5 gram is ignited in a platinum capsule for ten minutes in a muffle furnace. The temperature should not exceed 800° C., or there will be a loss of SO_3.

Loss in weight × 200 = loss on ignition (CO_2 + H_2O + organic).

Carbon Dioxide.—If necessary this may be estimated by treating 5 grams with hydrochloric acid in the absorption apparatus as described in (14).

(131) R. K. Meade (*Portland Cement*) recommends the use of a Shimer crucible. This consists of a platinum crucible provided with a water-jacketed stopper and reservoir for supplying water to the latter. From 1 to 3 grams of cement is placed in the crucible and covered with ignited asbestos. The crucible is heated by means of a bunsen burner after starting a flow of hot water through the stopper. The gas is aspirated through potash bulbs and calcium chloride tubes in the usual way. Finally the crucible is heated over a blast burner. The absorption apparatus is weighed; increase of weight equals CO_2. If the cement contains any unburnt carbonaceous material this causes an error. The carbon may be estimated by treating the cement with acid, filtering through asbestos, and then igniting residue in the crucible. For details see work cited.

(132) **Rapid Method for Estimation of Lime in Cement.**—Weigh 0·5 gram of cement into a dry wide-form 800-c.c. beaker, add about 10 c.c. of distilled water, and rotate to prevent setting, then add 20 c.c. of 10E HCl. Warm until solution is complete. Dilute to about 250 c.c. with warm water, boil for a few minutes, then exactly neutralise with 10E NH_4OH, using methyl orange as

indicator. To the boiling solution add 10 c.c. of $\frac{3E}{2}$ (concentrated) oxalic acid solution, boil for one minute, then add 70 c.c. of $\frac{3E}{5}$ ammonium oxalate solution. Boil for seven minutes, remove from heat, allow to settle, and filter through a 15-cm. rapid paper. Wash several times by decantation, using plenty of hot water; then transfer to filter paper and wash until a drop of the filtrate acidified with sulphuric acid does not decolourise water faintly tinted with permanganate. About 700 c.c. of wash water will be required; with practice, always using the same quantity of reagents, one can readily gauge the amount of warm water to use.

Remove the filter from the funnel, open and lay against the sides of the beaker in which precipitation was made. Wash ppt. from the paper into the beaker. Add 30 c.c. or sufficient 5E H_2SO_4, warm, titrate with standard permanganate, of which the strength in terms of CaO is known.

Number of c.c. used × factor × 200   CaO.

(133) The permanganate may be standardised against pure Iceland spar, or better against a "standard" cement, the lime in which had been carefully estimated by a gravimetric method. Pure recrystallised oxalic acid may also be employed.

A convenient strength is obtained by using

6 to 7 grams of permanganate per litre; the solution should be made some days before it will be required.

(134) **ANALYSIS OF GYPSUM, PLASTER, KEENE'S CEMENT, Etc.**—All these bodies consist of calcium sulphate in one form or another. The necessary estimations include silica + insoluble matter, alumina and iron oxide, lime, magnesia, sulphuric anhydride, and water.

(135) **Silica and Insoluble.**—Weigh out into a porcelain or platinum dish 1 gram of the finely powdered material. Add a little water and rotate to prevent setting. Add 10 c.c. 10E HCl and evaporate carefully to dryness. Take up with 25 c.c. of 10E HCl and water. Digest, if necessary: filter off through a small filter paper any insoluble matter, wash well, dry, ignite, and weigh.

Weight   100 = per cent. insoluble.

(136) Make the filtrate up to 500 c.c. and divide into two parts of 250 c.c. each.

Alumina + Ferric Oxide, Lime, and Magnesia.—In one portion estimate Al_2O_3, Fe_2O_3, CaO, and MgO by the ordinary methods.

(137) **Sulphuric Anhydride.**—Place the other portion of 250 c.c. in a fairly large beaker and bring to the boil; whilst gently boiling, add

20 c.c. of E $BaCl_2$ drop by drop. Allow ppt. to settle, filter, wash, ignite, and weigh.

Weight of ppt. × 0·3431 × 200 = per cent. SO_3.
Weight of ppt. × 0·583 × 200 = per cent. $CaSO_4$.

(138) **Water.**—Weigh 0·5 gram of the finely ground sample into a platinum crucible and heat in the oven for one hour at 100° C.

Loss in weight × 200 = per cent. moisture at 100° C.

(138*b*) Ignite the dried material from (138) over a bunsen burner or in the muffle furnace at a temperature of between 350° to 400° C. for half an hour, allow to cool in desiccator and weigh. Repeat to constant weight.

Loss in weight × 200 = per cent. water at 400° C.

(139) **ESTIMATION OF CAUSTIC LIME (CaO) IN BURNT LIME.**—The material under examination should be sampled, crushed, and finely powdered as rapidly as possible. The original sample should be stored in an air-tight receptacle, and the portion for analysis placed in a dry weighing tube.

(140) **Estimation of CaO.**—Transfer about 0·5 gram of the finely powdered sample into an Erlenmeyer flask containing about 250 c.c. of air-free distilled water. Boil gently for five

minutes, close the flask with a cork bearing a soda-lime tube, and allow to cool. When quite cold, titrate with $\frac{N}{10}$ HCl, using phenolphthalein as indicator; allow the flask to remain some time to see if the pink colouration returns.

$$\frac{\text{No. of c.c. used} \times \cdot 0028 \times 100}{\text{weight taken}} = \text{per cent. CaO.}$$

CHAPTER VI.

THE ANALYSIS AND EXAMINATION OF AGGREGATES, CEMENT - SAND MIXTURES, CONCRETE, ETC.

Although not strictly the work of the works-chemist, it sometimes falls to his lot to examine the materials intended for use as aggregates in concrete work, or the concrete itself. In connection with the latter work it may at once be pointed out that it is extremely difficult to obtain even partially satisfactory results unless the composition of either the materials forming the aggregate or the cement is known. Such work is generally only required in case of a failure or dispute as to the proportion of cement used, and the latter problem is complicated by the fact that the component parts are generally proportioned by measure and not by weight.

(141) **AGGREGATES.**—The material constituting the aggregate may consist of gravel, shingle, limestone, sandstone, or other natural rocks, burnt clay, slag, coke breeze, clinkers, etc., and sand.

The testing of sand will be dealt with specially.

All the other materials should be tested for soluble salts, "free" lime, sulphur, and organic matter, the latter two especially in the case of slag, coke breeze, and clinkers.

The processes of analysis already described may be employed.

The material should also be tested for dust, clay, etc., as described under sand (143). The size and proportion of material retained on a 3-inch, 2-inch, 1-inch, and ½-inch mesh sieve may also have to be determined. A full analysis will not be generally required except for purposes of comparison, when a sample of the concrete made therefrom has also to be examined. The processes of analysis employed will vary according to the class of material. But those described under limestones, clay, slag, etc., will be found generally applicable.

The suitability or otherwise of a material depends upon the class of work for which the concrete is required.

(142) For the purpose of correctly proportioning the various materials to be used in making concrete, it is necessary to ascertain the "percentage of voids" in an aggregate. This may be carried out in the laboratory as follows:—Take a graduated vessel, as large as possible, say 1,000 c.c., and fill up to the mark with the material to be tested, shaking down well, then from another graduated vessel pour in water also up to the

mark. The volume of water divided by 10 will give the percentage of voids in the material. If the material is at all absorbent it should be previously saturated with water.

(143) **SAND.** All materials, and especially sand, should be tested for soluble salts, clay, and dust. One process may be made to suffice. Weigh up as large a quantity of the well-mixed and dry material as can be conveniently handled, say 500 grams. Shake up with sufficient distilled water, preferably in a wide-mouthed glass jar or bottle, and allow to stand. The heavier material will quickly settle whilst any clay or dust will remain in suspension, sometimes for hours.

In the absence of clay or dust, decant a portion of the supernatant liquid, test reaction towards litmus, and then evaporate to dryness to test for presence of soluble salts. If ordinary tap water is used the content of total solids must be ascertained previously.

In the presence of clay or dust it will be necessary to filter.

The nature of the clay or dust may be ascertained by again agitating the liquid and pouring off a portion whilst the lighter particles are still in suspension. Evaporate to dryness and analyse as under clay (19). A mechanical analysis of the sand may be performed as under (18).

The presence of clay renders a material quite unfit for concrete constructional purposes.

(144) **The Examination of Concrete.**—The examination of a sample of set concrete entails the employment of both mechanical and chemical processes.

A large sample, at least 1,000 grams, should be broken up carefully with an iron pestle and mortar, avoiding as far as possible any disintegration of the larger portions forming the aggregate. Separate the latter by hand or by passing through a sieve, and then remove as completely as possible by scraping with a knife, and stiff brush any adherent cement and sand. The proportion of the larger nodules may be roughly ascertained by weighing.

With some materials it may be impossible to perform a mechanical separation, in which case the whole of the sample should be crushed and powdered, if necessary, in a mechanical crusher and mill, and a chemical analysis entirely relied upon. In any case, powder the portion consisting of cement and sand to pass a 100-mesh sieve, and reserve for analysis. Remove any particles of iron or steel which may have become detached from the mortar or mill by means of a magnet and ignore. In the case of ferro-concrete, a portion of the reinforcement may be embedded in th mass; this should be removed before weighin out the sample.

The sample may then be analysed, first, a follows:—Soluble salts (18*b*), silica and insolubl (117) and (127), alumina and ferric oxide (118) lime (119) and magnesia (119), loss on ignition (130).

If the aggregate is known to consist of sand or gravel entirely insoluble in 10E HCl and 3E Na_2CO_3 as used in the methods of analysis described, the percentage so found may be stated as "aggregate." If any of the known aggregate has been removed by mechanical means this must be allowed for, and the original approximate composition obtained by calculation. In this case the soluble silica, alumina, and ferric oxide, lime and magnesia will constitute approximately to the cement used. The loss on ignition will include the original "loss on ignition" of the cement, which may be ignored, water of hydration, carbon dioxide, and organic matter. Should there be any indications that the latter includes coal dust, coke dust, oil, tar, etc., a special method, such as extraction with ether, benzene, alcohol, etc., will be rendered necessary, also a microscopic examination.

In the case of a "failure," it will be very necessary to carry out an exhaustive qualitative analysis, and also from the quantitative analysis to calculate as far as possible the original composition of the cement used. From the results of an analysis it may be possible together with the data given as to average specific gravity or weight per unit volume of the materials used to calculate the parts by volume taken and thus to ascertain whether the concrete was made in accordance with the specification.

When the aggregate contains a material soluble in the reagents used, such as limestone, unless it can be satisfactorily separated by mechanical means, the problem becomes considerably complicated, and in fact, unless the composition of both aggregate and cement are known, practically unsolvable with any degree of accuracy.

The aim of the cement works' chemist engaged upon such work should be to prove as far as possible that the fault does not lie with the cement used. To this end it will be useful, perhaps, to estimate the sulphuric anhydride, and magnesia, although the presence of these in the aggregate may render the data useless.

In calculating the composition of a concrete mixture by volume from per cent. by weight, the following data will be found useful :– 1 cubic foot of coarse gravel weighs about 95 lbs., 1 cubic foot of sand about 100 to 105 lbs., 1 cubic foot of cement weighs about 90 lbs. Thus a 4-2-1 mixture by volume of gravel-sand-cement would analyse 56 to 57 per cent. gravel, 30 per cent. sand, 13 to 14 per cent. cement.

CHAPTER VII.

NOTES ON CEMENT TESTING.

For the purposes of the British Standard Specification it is necessary to carry out the following tests:—

(*a*) Fineness, (*b*) specific gravity, (*c*) chemical composition, (*d*) tensile strength (neat cement), (*e*) tensile strength (cement and sand), (*f*) setting time, (*g*) soundness.

With reference to (*a*), (*d*), (*e*), and (*g*), certain instructions are given in the specification for the carrying out of these tests. The following hints, largely supplied by an expert gauger, are only intended to amplify the official instructions, and in the case of (*b*) and (*g*) and the non-official tests to act as a guide for the less experienced.

(*a*) *Fineness.*—Weigh out 100 grams of cement, free from air-set lumps, sieve as per official instructions, brush residue on to a tared watch-glass and weigh.

A "linen-counter" will be found useful for checking and examining the meshes of the sieves.

(*b*) *Specific Gravity* (Blount's flask).—Weigh out

50 grams of cement, place on a sheet of glazed paper, note temperature and measure out 50 c.c. of "paraffin oil," which has been kept over fused calcium chloride, carefully run into the perfectly dry flask by means of a pipette. Stand the flask on another piece of glazed paper and carefully transfer the weighed cement, by means of a short-stemmed funnel into the flask. This should be done gradually, the bottle being rotated and lightly rapped on the bench to disengage air-bubbles, between each addition of cement. Any stoppage in the neck of flask or funnel should be removed by means of a clean, stout knitting needle or other suitable instrument.

When all the cement has been transferred to the bottle, remove the funnel, replace stopper, and agitate contents as long as air bubbles arise. Allow to stand for a short time and read off volume of oil displaced on neck of flask.

Then

$$\text{Specific gravity} = \frac{50}{\text{c.c. displaced}}.$$

It is important that the temperature should not alter during the carrying out of the operation; if so the flask must be brought to the original temperature by standing in water.

With the Anderson bottle, 150 grams of cement and 200 c.c. of oil are used, the neck of the bottle being larger, it is somewhat more easily filled and the air removed than in the case of the smaller

(*Blount*) *flask*. To clean flask, shake out contents as completely as possible, removing any remaining traces with oil; if allowed to settle the oil may be used repeatedly.

In the absence of a cement gravity flask, the specific gravity may be determined by using an ordinary pyknometer or specific gravity bottle, preferably with a wide neck.

Ascertain the specific gravity of the oil in the usual way (see 69). Introduce a weighed portion of the cement into the empty bottle, fill with the oil and weigh. The specific gravity of the cement may then be calculated from the formula

$$\text{Specific gravity} = \frac{W}{W - W_1} \times L.$$

W = weight of cement in air, W_1 = weight of cement in oil, and L = specific gravity of the oil at the same temperature.

(*c*) *Chemical Composition.* The requirements of the specification have already been referred to (see Chap. V.).

(*d*) and (*e*) *Tensile Strength.*—Before attempting to gauge the cement for either tensile or compression tests, the "initial setting time" (*f*) should be determined, at or near the temperature at time of gauging. If quick setting, each briquette should be gauged separately; if slow setting, the cement for three or more moulds may be gauged at one operation.

For each neat briquette weigh up 200 grams of cement. For each sand briquette

150 grams of cement.
50 „ standard sand.

The proper proportion of water to be used may be ascertained by trial.

(*f*) *Setting Time.*—In order to ascertain the correct proportion of water to use, weigh up 400 grams of cement and gauge with 80 c.c. of water, adding successive quantities of 10 c.c. until the pat is of correct consistency. Discard this experimental test piece, or at any rate, repeat using correct quantity of water, and complete the gauging well within the limit of time of initial set.

For works' purposes the setting time may be ascertained upon a pat of the neat cement, using the Gilmore needle or the thumb nail. Attention should be paid to the temperature and atmospheric conditions during the test.

(*g*) The official test for soundness is by the Le Chatelier method; full instructions for carrying out will be found in the standard specification.

For contract and works' purposes the following qualitative tests will be found very useful:—

Gauge up sufficient neat cement with the correct proportion of water to make eight pats on pieces of clean glass.

(1) *Plunge Test.*—Place two pats as soon as gauged in clean cold water. These should set

hard and show no signs of cracking or disintegration.

(2) *Faija Test.*—Place two of the freshly gauged pats in the moist steam at 100° - 105° F., preferably in the bath especially constructed for this test, until thoroughly set. Then immerse in the water kept at 115° to 120° F. for the remainder of the twenty-four hours.

The pat should not leave the glass or show any sign of cracking or blowing.

(3) Allow the remaining pats to set in air, and when set, place two in cold water, and bring to the boil gradually and keep boiling for six hours. This is rather a severe test, but the pats should not show any signs of cracking, even if they leave the glass. The other two pats are kept for reference purposes and for colour and general appearance.

Compression Tests.—The compression test, although not yet officially adopted, is being largely used on the principal works, and by important private firms and public bodies.

Owing to the larger quantity required for filling the cubical moulds, special attention should be given to the gauging and to the careful mixing of the sand and cement.

For each neat cube ($2\frac{3}{4}$ inches) take 800 grams of cement. For each sand cube ($2\frac{3}{4}$ inches) take 750 grams of cement and 250 grams of standard sand.

USEFUL DATA FOR TESTING ROOM.

To convert—

Lbs. per sq. inch into kilos per sq. centimetre	by ·0703
Kilos per sq. centimetre into lbs. per sq. inch	by 14·223
Mesh per sq. centimetre into mesh per sq. inch	by 6·45
Mesh per sq. inch into mesh per sq. centimetre	by ·155
Grams into grains	by 15·43235
Grains into grams	by ·0648
Degrees Centigrade into degrees Fahrenheit (F.)	$\frac{9}{5}$ and add 32.
Degrees Fahrenheit into degrees Centigrade (C.)	subtract 32 and by $\frac{5}{9}$.

APPENDIX.

APPENDIX.

EXAMPLES OF CALCULATIONS OCCURRING DURING ANALYSIS.

(1A) **Clay Analysis.**—Conversion of insoluble into felspar (21).

Found. Insol. 12·88 per cent. containing 1·71 per cent. Al_2O_3 :

$$
\begin{array}{lll}
\text{then } 1{\cdot}71 \times 3{\cdot}5 & = 5{\cdot}98 & \text{per cent. } SiO_2 \\
\phantom{\text{then }} 1{\cdot}71 \times 0{\cdot}9 & = 1{\cdot}54 & \phantom{\text{per cent. }} ,, \quad K_2O \\
\phantom{\text{then }} Al_2O_3 \text{ found} & 1{\cdot}71 & \\
\hline
 & 9{\cdot}23 &
\end{array}
$$

12·88 − 9·23 = 3·65 per cent. of quartz.

Report as follows :

$$
\text{Insoluble matter } 12{\cdot}88 \left\{
\begin{array}{ll}
\left.\begin{array}{ll}
SiO_2 & = 5{\cdot}98 \\
Al_2O_3 & = 1{\cdot}71 \\
K_2O & = 1{\cdot}54
\end{array}\right\} & \text{felspar.} \\
\begin{array}{ll}
\text{Quartz} & = 3{\cdot}65
\end{array} & \\
\hline
 \quad 12{\cdot}88 &
\end{array}
\right.
$$

(2A) To ascertain proportions of limestone and shale to obtain desired mixture.

Ultimate Analysis.

	Limestone.	**Shale.**
SiO_2	5·96	49·74
Al_2O_3	2·00	15·98
Fe_2O_3	0·90	8·00
CaO	49·38	9·08
MgO	1·12	4·03
Loss on ignition	40·12	10·04
Alkalies and loss	0·52	3·13
	100·00	100·00

Calculation.

Limestone.

SiO_2 = 5·96 × 2·8 = 16·688
Al_2O_3 = 2·00 × 1·1 = 2·200

18·888*

Shale.

SiO_2 = 49·74 × 2·8 = 139·272
Al_2O_3 = 15·98 × 1·1 = 17·578

157·850

CaO = 49·380
− 18·888*

y = 30·492

157·85
− CaO in shale 9·08

x = 148·77

$$\text{Then } \frac{148{\cdot}77 \times 100}{30{\cdot}492} = 487{\cdot}9.$$

Therefore 100 parts of shale should require 487·9 parts of limestone, or

Limestone 4·8 parts.
Shale 1·0 part.

(3A) To produce mixture containing 75 per cent. $CaCO_3$; same materials.

CaCO in limestone = 88·18 per cent.
Per cent. required = 75·00 ,,
Shale required 13·18 parts.

Per cent. required = 75·00
$CaCO_3$ in shale 16·21
Limestone required 58·79 parts.

or Limestone 4·4 parts.
Shale 1·0 part.

(4A) **Analysis of Slurry** (54).

Example of calculation :

$CaCO_3$ = 75·2 per cent. = CO_2 33·08 per cent. (B).
Loss on ignition = 33·80 per cent. (A).

Then 33·80
−33·08

= 0·72 loss due to organic matter (C).
and 100 − 0·72 99·28 (D).

$$\frac{75{\cdot}2 \times 100}{99{\cdot}28} = 75{\cdot}74 \text{ calculated } CaCO_3.$$

(5A) *Calculated CaO* (55).

Lime in residue after ignition = 42·4 per cent.
Loss on ignition = 33·8 ,,

$\frac{42\cdot4 \times 100}{100 - 33\cdot8}$ = 64 per cent. calculated CaO in clinker.

(6A) Directions for making up E Solutions.

Hydrochloric acid (sp. gr. 1·16) = 10 E HCl.
,, ,, (,, 1·08) = 5 E HCl.
,, 200 c.c. of 5 E HCl diluted to 1 litre = E HCl.
Nitric acid (sp. gr. 1·42) = 16 E HNO_3.
,, (,, 1·16) = 5 E HNO_3.
,, 200 c.c. of 5 E HNO_3 diluted one litre = E HNO_3.
Sulphuric acid (sp. gr. 1·842) = 36 E H_2SO_4.
Acetic acid (glacial) 17 E $C_2H_4O_2$.
,, 294 c.c. 17 E diluted to 1 litre = 5 E $C_2H_4O_2$.
Oxalic acid 94·5 grams dissolved and diluted to make 1 litre = $\frac{3}{2}$ E $C_2H_2O_4$.
Ammonium hydrate (·880) = 20 E NH_4OH.
,, ,, equal volumes of 20 E and distilled water = 10 E NH_4OH.
,, oxalate 42·6 grams dissolved and diluted to 1 litre = $\frac{3}{5}$ E$(NH_4)_2C_2O_4$.

Ammonium carbonate 196·7 grams dissolved in 333·3 c.c. of 5 E NH_4OH and diluted to litre = 5 E Am_2CO_3.

Sodium hydrate 200 grams dissolved to make 1 litre = 5 E NaOH.

,, carbonate 429 grams of crystals to make 1 litre = 3 E Na_2CO_3.

,, hydrogen phosphate 119·3 grams of crystals dissolved to make 1 litre = $\frac{2}{3}$ E Na_2HPO_4.

,, sulphite 252 grams of crystals to make 1 litre = 4 E Na_2SO_3.

,, acetate 544 grams of crystals to make 1 litre = 4 E $NaC_2H_3O_2$.

Potassium hydrate 280 grams to make 1 litre 5 E KOH.

,, iodide 166 grams to make 1 litre = E KI.

,, chromate 97·25 grams to make 1 litre = E K_2CrO_4.

,, ferrocyanide 105·5 grams to make 1 litre = E $K_4FeC_6N_6$.

,, ferricyanide 109·7 grams to make 1 litre E $K_3FeC_6N_6$.

,, sulphocyanide 97 grams to make 1 litre = E KCNS.

Bromine water—water at 15·5° C. saturated with Br = $\frac{E}{2}$ Br water.

Barium chloride 122 grams dissolved to make 1 litre E $BaCl_2$.

Silver nitrate 170 grams dissolved to make 1 litre = E $AgNO_3$.

,, 34 grams dissolved to make 1 litre $\frac{E}{5}$,,

Mercuric chloride 24·2 grams dissolved to make 1 litre $\frac{2E}{5}$ $HgCl_2$.

Copper sulphate 124·75 grams of crystals to make 1 litre = E $CuSO_4$.

Platinic chloride 49·3 grams of Pt converted into $PtCl_4$ and diluted to 1 litre E $PtCl_4$.

Magnesia mixture 68 grams $MgCl_2 6H_2O$ in about 500 c.c. H_2O, add 165 grams AmCl, 300 c.c. 5E NH_4OH and dilute to 1 litre = E magnesia mixture.

Ammonium molydate.—Dissolve 90 grams in 700 c.c. of water, allow to settle and decant; dilute to 1 litre.

Fusion mixture—

106 grams of dry Na_2CO_3
138 ,, ,, K_2CO_3

Well mixed and ground together.

Mixture for Hundeshagen's method (63) for sulphur in fuels—

MgO 2 parts by weight.
K_2CO_3 1 part ,,

Dried and ground together.

Copper sulphate pumice for absorption (14) of HCl H_2O ·

Soak some pieces of pumice stone free from dust in a saturated solution of copper sulphate. When thoroughly saturated, dry and ignite until white. Store in a well-stoppered bottle.

PREPARATION OF STANDARD SOLUTIONS.

(7A) **Normal Sodium Carbonate**, $N.Na_2CO_3$.

Dissolve exactly 53 grams of Na_2CO_3 (prepared by igniting bicarbonate) in distilled water and make up to 1,000 c.c.

1 c.c. = ·053 gram Na_2CO_3
= ·022 ,, CO_2.

(8A) **Deci-normal** Na_2CO_3 $\frac{N}{10}$ Na_2CO_3.

Dissolve 5·3 grams in 1,000 c.c. of distilled water.

1 c.c. = ·0053 gram Na_2CO_3.

(9A) **Normal Sulphuric Acid**, $N.H_2SO_4$.

Dilute about 30 c.c. of conc. H_2SO_4 (sp. gr. 1·840) to 1 litre. Add the acid to less than 1,000 c.c. of water, and when cool, titrate against $N.Na_2CO_3$, using methyl orange as indicator. Then measure the solution and dilute to correct volume.

1 c.c. = ·049 gram H_2SO_4.

(10A) **Deci-normal Sulphuric Acid** $\frac{N}{10}$ H_2SO_4.

Dilute 100 c.c. of normal H_2SO_4 to 1,000 c.c. and standardise against $\frac{N}{10}$ Na_2CO_3.

1 c.c. = ·0049 gram H_2SO_4.

(11A) **Normal Hydrochloric Acid.** N.HCl.

Dilute 150 c.c. of pure HCl (sp. gr. 1·16) to 1,000 c.c., titrate with $N.Na_2CO_3$ and correct accordingly.

1 c.c. = ·0365 gram HCl.

(12A) **Deci-normal HCl** $\frac{N}{10}$ HCl.

Dilute 100 c.c. of N.HCl to 1,000 c.c. with distilled water.

1 c.c. = ·00365 gram HCl.

(13A) **Normal Sodium Hydrate.** N.NaOH.

Dissolve about 44 grams of stick NaOH, free from carbonate, in about 1,000 c.c. of distilled water. Titrate, when cool, against normal H_2SO_4 and dilute accordingly; use methyl orange or phenolphthalein as indicator.

1 c.c. = ·040 gram NaOH.

(14A) Deci-normal $NaOH$.

Dilute 100 c.c. $N.NaOH$ to 1,000 c.c. with distilled water.

1 c.c. = ·0040 gram $NaOH$.

(15A) Normal Potassium Hydrate. $N.KOH$.

Dissolve 56 grams in less than 1,000 c.c. of water, titrate against $N.H_2SO_4$, and dilute accordingly. For alcoholic KOH use 90 per cent. alcohol.

1 c.c. = ·056 gram KOH.

(16A) Deci-normal KOH. $\frac{N}{10}$ KOH.

Dilute 100 c.c. of normal KOH to 1,000 c.c. and standardise.

1 c.c. ·0056 gram KOH.

(17A) Deci-normal Potassium Permanganate. $\frac{N}{10}$ $K_2Mn_2O_8$.

Dissolve 3·156 grams of potassium permanganate in distilled water and dilute to 1,000 c.c. Titrate with pure iron wire, ferrous ammonium sulphate, or oxalic acid.

1 c.c. $\frac{N}{10}$ $K_2Mn_2O_8$ = ·0056 gram Fe.
= ·0072 „ FeO.
= ·0080 „ Fe_2O_3.

(18A) Deci-normal Potassium Bichromate. $\frac{N}{10}$ $K_2Cr_2O_7$.

Dissolve 4·913 grams of fused crystals in water and make up to 1,000 c.c.

1 c.c. = ·0056 gram Fe.

This solution requires standardisation after a time. Use potassium ferricyanide on a spotting tile as indicator.

(19A) Deci-normal Silver Nitrate.

$\frac{N}{10}$ $AgNO_3$.

Dissolve 16·998 grams in distilled water and make up to 1,000 c.c.

1 c.c. = ·003546 gram Cl.

(20A) Standard Iodine Solution.

Hubl.—25 grams iodine in 500 c.c. of alcohol. 25 grams of mercuric chloride in 500 c.c. of alcohol. Use equal parts.

Hanus.—13 grams of iodine in 1 litre of glacial acetic acid. Then add 3 c.c. of bromine.

INDICATORS.

(21A) *Litmus Solution.*—Digest the solid with distilled water for several hours. Decant or filter, render neutral by means of acetic acid or ammonia. Store in a bottle with access of air.

Methyl Orange.—Dissolve about 1 gram of the solid in distilled water and make up to 1,000 c.c.

Phenolphthalein.—Dissolve a little of the solid in alcohol and dilute with alcohol and water.

(22A) TABLE OF ATOMIC WEIGHTS OF PRINCIPAL ELEMENTS OCCURRING IN CEMENT WORKS ANALYSIS.

Aluminium	Al	27·10	Nitrogen	N	14·01
Barium	Ba	137·37	Oxygen	O	16·00
Calcium	Ca	40·07	Phosphorus	P	31·04
Carbon	C	12·00	Platinum	Pt	195·2
Chlorine	Cl	35·46	Potassium	K	39·10
Copper	Cu	63·57	Silicon	Si	28·30
Hydrogen	H	1·008	Silver	Ag	107·88
Iron	Fe	55·84	Sodium	Na	23·0
Iodine	J	126·92	Sulphur	S	32·07
Magnesium	Mg	24·32	Titanium	Ti	48·10
Manganese	Mn	54·93			

(23A) FACTORS FOR USE IN ANALYSIS.

Required.	Known.	Factor.
CaO	$CaSO_4$	0·41200
CaO	$CaCO_3$	0·56030
$CaCO_3$	CaO	1·77847
C	CO_2	0·27272
Fe	Fe_2O_3	0·69939
Fe_2O_3	Fe	1·42979
H_2SO_4	$BaSO_4$	0·42030
S	,,	0·13743
SO_3	,,	0·34310
$CaSO_4$	,,	0·58336
KCl	K_2PtCl_2	0·30673
K_2O	K_2PtCl_6	0·19376
K_2O	KCl	0·63170
Na_2O	$NaCl$	0·53027
MgO	$Mg_2P_2O_7$	0·36206
MnO	Mn_3O_4	0·93006
P_2O_5	$Mg_2P_2O_7$	0·63793

Tables (22A) and (23A) have been compiled from the Table of International Atomic Weights (1916).

(24A) LIME IN CEMENT (119).

USING 0·5 GRAM OF CEMENT.

Milligrams in $CaSO_4$.	Per cent. CaO.	Milligrams in $CaSO_4$.	Per cent. CaO.	Milligrams in $CaSO_4$.	Per cent. CaO.
694	57·18	726	59·82	758	62·45
695	57·26	727	59·90	759	62·54
696	57·35	728	59·97	760	62·62
697	57·43	729	60·06	761	62·70
698	57·51	730	60·15	762	62·78
699	57·59	731	60·23	763	62·87
700	57·68	732	60·31	764	62·95
701	57·77	733	60·39	765	63·03
702	57·84	734	60·48	766	63·11
703	57·92	735	60·56	767	63·20
704	58·00	736	60·64	768	63·28
705	58·09	737	60·72	769	63·36
706	58·17	738	60·81	770	63·44
707	58·25	739	60·89	771	63·53
708	58·34	740	60·97	772	63·61
709	58·42	741	61·05	773	63·69
710	58·50	742	61·14	774	63·77
711	58·58	743	61·22	775	63·86
712	58·66	744	61·30	776	63·94
713	58·75	745	61·38	777	64·02
714	58·83	746	61·47	778	64·10
715	58·91	747	61·55	779	64·18
716	58·99	748	61·63	780	64·27
717	59·08	749	61·71	781	64·35
718	59·16	750	61·80	782	64·43
719	59·24	751	61·88	783	64·51
720	59·32	752	61·96	784	64·6
721	59·41	753	62·04	785	64·68
722	59·49	754	62·12	786	64·76
723	59·57	755	62·21	787	64·84
724	59·65	756	62·29	788	64·93
725	59·74	757	62·37	789	65·01

(25A) TABLE FOR ESTIMATION OF MgO IN CEMENT (126).

USING 0·5 GRAM OF CEMENT.

Weight of $Mg_2P_2O_7$.	MgO per cent	Weight of $Mg_2P_2O_7$.	MgO per cent	Weight of $Mg_2P_2O_7$.	MgO per cent
Gram		Gram		Gram.	
·0080	0·57	·0195	1·41	·0310	2·24
·0085	0·61	·0200	1·44	·0315	2·28
·0090	0·65	·0205	1·48	·0320	2·31
·0095	0·68	·0210	1·52	·0325	2·35
·0100	0·72	·0215	1·55	·0330	2·38
·0105	0·76	·0220	1·59	·0335	2·42
·0110	0·79	·0225	1·62	·0340	2·46
·0115	0·83	·0230	1·66	·0345	2·49
·0120	0·86	·0235	1·70	·0350	2·53
·0125	0·90	·0240	1·73	·0355	2·57
·0130	0·94	·0245	1·77	·0360	2·60
·0135	0·97	·0250	1·81	·0365	2·64
·0140	1·01	·0255	1·84	·0370	2·67
·0145	1·04	·0260	1·88	·0375	2·71
·0150	1·08	·0265	1·91	·0380	2·75
·0155	1·12	·0270	1·95	·0385	2·78
·0160	1·15	·0275	1·99	·0390	2·82
·0165	1·19	·0280	2·02	·0395	2·86
·0170	1·23	·0285	2·06	·0400	2·89
·0175	1·26	·0290	2·09	·0405	2·93
·0180	1·30	·0295	2·13	·0410	2·96
·0185	1·33	·0300	2·17	·0415	3·00
·0190	1·37	·0305	2·20	·0420	3·04

(26A) TABLE FOR ESTIMATION OF SO_3 IN CEMENT (128).

USING 0·5 GRAM OF CEMENT.

Weight of $BaSO_4$	SO_3 per cent.	$CaSO_4$ per cent	Weight of $BaSO_4$	SO_3 per cent	$CaSO_4$ per cent
·0100	0·68	1·16	·0205	1·40	2·39
·0105	0·72	1·22	·0210	1·44	2·45
·0110	0·75	1·28	·0215	1·47	2·51
·0115	0·78	1·31	·0220	1·50	2·57
·0120	0·82	1·40	·0225	1·54	2·63
·0125	0·05	1·46	·0230	1·57	2·68
·0130	0·89	1·52	·0235	1·61	2·74
·0135	0·92	1·57	·0240	1·64	2·80
·0140	0·96	1·63	·0245	1·68	2·86
·0145	0·99	1·69	·0250	1·71	2·92
·0150	1·02	1·75	·0255	1·74	2·98
·0155	1·06	1·81	·0260	1·78	3·03
·0160	1·09	1·87	·0265	1·81	3·09
·0165	1·13	1·93	·0270	1·85	3·15
·0170	1·16	1·98	·0275	1·88	3·20
·0175	1·20	2·04	·0280	1·92	3·26
·0180	1·23	2·10	·0285	1·95	3·32
·0185	1·26	2·16	·0290	1·99	3·38
·0190	1·30	2·22	·0295	2·02	3·44
·0195	1·33	2·28	·0300	2·05	3·50
·0200	1·37	2·33			

(27A) TABLES FOR USE WITH SLATER'S CALCIMETER.

Temp. of paraffin. Deg. C.	Weight of Sample in Milligrams.			Temp. of paraffin. Deg. C.	Weight of Sample in Milligrams.			Temp. of paraffin. Deg. C.	Weight of Sample in Milligrams.		
	A 70–80 per cent.	B 80–91 per cent.	C 91–100 per cent.		A 70–80 per cent	B 80–91 per cent.	C 91–100 per cent		A 70–80 per cent.	B 80–91 per cent.	C 91–100 per cent.
5·0	922	807	692	14·5	881	771	661	21·0	839	734	630
5·5	920	805	690	15·0	879	769	659	21·5	836	732	628
6·0	918	803	688	15·5	877	767	658	25·0	831	730	626
6·5	916	801	686	16·0	875	766	656	25·5	832	728	624
7·0	913	799	685	16·5	873	764	655	26·0	830	726	623
7·5	911	797	683	17·0	871	762	653	26·5	828	725	621
3·0	909	795	682	17·5	869	761	652	27·0	826	723	620
8·5	907	794	680	18·0	867	759	650	27·5	824	721	618
9·0	905	792	679	18·5	865	757	649	28·0	822	719	616
9·5	903	790	677	19·0	863	755	647	28·5	820	718	615
10·0	901	788	676	19·5	860	753	615	29·0	818	716	614
10·5	899	787	674	20·0	858	751	644	29·5	816	714	612
11·0	896	785	672	20·5	856	749	642	30·0	814	712	610
11·5	894	783	671	21·0	854	747	641	30·5	811	710	608
12·0	892	781	669	21·5	852	745	639	31·0	808	707	606
12·5	890	779	668	22·0	850	743	638	31·5	805	704	604
13·0	888	777	666	22·5	848	741	636	32·0	802	702	602
13·5	886	775	661	23·0	845	739	631	32·5	799	699	599
14·0	883	773	662	23·5	842	737	632	33·0	796	697	597

Note.—This Table should only be taken as a guide when standardising a new instrument.

(28A) TABLE FOR USE WITH SLATER'S CALCIMETER.

ESTIMATION OF $CaCO_3$ IN LIMESTONE.

Reading.	Weight taken A — $CaCO_3$ As reading.	Weight taken B (·875 of A) — $CaCO_3$	Weight taken C (·75 of A) — $CaCO_3$
70·00	—	80·00	93·34
70·25	—	80·28	93·67
70·50	—	80·57	94·00
70·75	—	80·85	94·34
71·00	—	81·14	94·67
71·25	—	81·42	95·00
71·50	—	81·71	95·34
71·75	—	82·00	95·67
72·00	—	82·28	96·00
72·25	—	82·57	96·34
72·50	—	82·85	96·67
72·75	—	83·14	97·00
73·00	—	83·42	97·34
73·25	—	83·71	97·67
73·50	—	84·00	98·00
73·75	—	84·28	98·34
74·00	—	84·57	98·67
74·25	—	84·85	99·00
74·50	—	85·14	99·34
74·75	—	85,42	99·67
75·00	—	85·71	100·00
75·25	—	86·00	
75·50	—	86·28	
75·75	—	86·57	
76·00	—	86·85	
76·25	—	87·14	
76·50	—	87·42	
76·75	—	87·71	
77·00	—	88·00	
77·25	—	88·28	
77·50	—	88·57	
77·75	—	88·85	
78·00	—	89·14	
78·25	—	89·42	
78·50	—	89·71	
78·75	—	90·00	
79·00	—	90·28	
79·25	—	90·57	
79·50	—	90·85	
79·75	—	91·14	
80·00	—	91·42	

(29A) TABLE OF CORRECTIONS TO BE ADDED TO CALCIMETER READING FOR VARYING AMOUNTS OF ORGANIC MATTER IN SLURRY.

RESULT = CALD. $CaCO_3$ (54).

Excess "Loss" over CO_2	Calcimeter Reading.									
	69·20 to 69·83	69·88 to 70·51	70·55 to 71·18	71·23 to 71·86	71·90 to 72·55	72·58 to 73·21	73·25 to 73·89	73·92 to 74·55	74·59 to 75·22	75·27 to 75·92
1·50	1·06	1·07	1·08	1·09	1·10	1·11	1·12	1·13	1·14	1·15
1·60	1·13	1·14	1·15	1·16	1·17	1·18	1·19	1·20	1·21	1·22
1·70	1·20	1·21	1·23	1·24	1·25	1·26	1·27	1·28	1·29	1·30
1·80	1·28	1·29	1·30	1·31	1·33	1·34	1·35	1·36	1·37	1·38
1·90	1·35	1·36	1·37	1·39	1·40	1·41	1·42	1·43	1·44	1·45
2·00	1·42	1·43	1·44	1·46	1·47	1·49	1·50	1·52	1·53	1·54
2·10	1·49	1·50	1·51	1·53	1·54	1·56	1·57	1·59	1·60	1·61
2·20	1·56	1·58	1·59	1·61	1·62	1·64	1·65	1·67	1·68	1·69
2·30	1·63	1·65	1·66	1·68	1·69	1·71	1·73	1·74	1·76	1·77
2·40	1·70	1·72	1·74	1·76	1·77	1·79	1·82	1·83	1·84	1·85
2·50	1·78	1·80	1·82	1·83	1·85	1·87	1·89	1·90	1·92	1·94
2·60	1·85	1·87	1·89	1·90	1·92	1·94	1·96	1·97	1·98	1·99
2·70	1·93	1·95	1·97	1·98	1·99	2·01	2·03	2·05	2·07	2·09
2·80	2·00	2·02	2·04	2·05	2·07	2·09	2·11	2·13	2·15	2·17
2·90	2·08	2·10	2·12	2·13	2·15	2·17	2·19	2·21	2·23	2·25
3·00	2·15	2·17	2·19	2·21	2·23	2·25	2·27	2·29	2·31	2·33
3·10	2·23	2·25	2·27	2·29	2·31	2·33	2·35	2·37	2·39	2·41
3·20	2·30	2·32	2·34	2·36	2·39	2·41	2·43	2·45	2·47	2·49
3·30	2·37	2·39	2·41	2·43	2·46	2·48	2·51	2·53	2·55	2·57
3·40	2·44	2·47	2·49	2·51	2·53	2·56	2·59	2·61	2·63	2·65
3·50	2·52	2·55	2·57	2·59	2·61	2·64	2·67	2·69	2·71	2·73
3·60	2·60	2·62	2·65	2·67	2·69	2·72	2·75	2·77	2·79	2·82
3·70	2·68	2·70	2·72	2·74	2·77	2·80	2·82	2·85	2·87	2·90
3·80	2·75	2·77	2·80	2·82	2·85	2·88	2·90	2·93	2·95	2·98
3·90	2·83	2·85	2·87	2·90	2·93	2·96	2·98	3·01	3·04	3·06
4·00	2·90	2·92	2·95	2·98	3·01	3·04	3·06	3·09	3·11	3·14
4·10	2·97	2·99	3·01	3·04	3·09	3·12	3·14	3·17	3·19	3·22
4·20	3·04	3·06	3·09	3·11	3·17	3·20	3·22	3·24	3·27	3·30
4·30	3·12	3·14	3·17	3·19	3·25	3·28	3·30	3·32	3·35	3·38
4·40	3·20	3·22	3·25	3·27	3·33	3·36	3·38	3·40	3·42	3·44
4·50	3·28	3·30	3·33	3·35	3·41	3·43	3·46	3·48	3·50	3·52

In the first column find the Excess "Loss on ignition" over CO_2, then in a line with it under the given calcimeter reading will be found the figure to be added in order to obtain "Calculated $CaCO_3$."

(30A) TABLE OF
PRESSURE OF AQUEOUS VAPOUR.

Temperature. Degrees C	Pressure in mm of mercury	Temperature. Degrees C.	Pressure in mm of mercury.	Temperature. Degrees C	Pressure in mm. of mercury	Temperature Degrees C.	Pressure in mm of mercury	Temperature. Degrees C.	Pressure in mm. of mercury
5·0	6·5	9·0	8·6	13·0	11·2	17·0	14·4	21·0	18·5
5·5	6·8	9·5	8·9	13·5	11·5	17·5	14·9	21·5	19·1
6·0	7·0	10·0	9·2	14·0	11·9	18·0	15·4	22·0	19·7
6·5	7·2	10·5	9·5	14·5	12·3	18·5	15·8	22·5	20·3
7·0	7·5	11·0	9·8	15·0	12·7	19·0	16·3	23·0	20·9
7·5	7·8	11·5	10·1	15·5	13·1	19·5	16·9	23·5	21·5
8·0	8·0	12·0	10·5	16·0	13·5	20·0	17·4	24·0	22·2
8·5	8·3	12·5	10·8	16·5	14·0	20·5	17·9	24·5	22·9

(31A) TEMPERATURE CORRECTION TABLE

0°C.	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23
77·4	78·71	79·11	79·40	79·70	80·00	80·30	80·59	80·89	81·18	81·48	81·68	82·07	82·37	82·67	82·97	83·37	83·67	83·97	84·36
77·6	78·91	79·31	79·60	79·90	80·21	80·50	80·79	81·09	81·39	81·68	81·88	82·27	82·57	82·87	83·17	83·57	83·87	84·17	84·56
77·8	79·11	79·51	79·80	80·10	80·41	80·70	80·99	81·30	81·59	81·88	82·08	82·47	82·77	83·07	83·38	83·77	84·07	84·38	84·76
78·0	79·32	79·71	80·01	80·31	80·61	80·90	81·19	81·50	81·79	82·08	82·28	82·67	82·97	83·28	83·58	83·97	84·27	84·58	84·96
78·2	79·52	79·92	80·21	80·51	80·81	81·10	81·30	81·70	81·99	82·29	82·48	82·88	83·17	83·48	83·78	84·17	84·48	84·78	85·16
78·4	79·72	80·12	80·41	80·71	81·01	81·31	81·50	81·90	82·21	82·49	82·68	83·08	83·38	83·68	83·98	84·38	84·68	84·98	85·37
78·6	79·92	80·32	80·61	80·91	81·22	81·51	81·70	82·10	82·41	82·69	82·89	83·28	83·58	83·88	84·18	84·58	84·88	85·18	85·57
78·8	80·12	80·52	80·81	81·01	81·42	81·71	81·90	82·31	82·61	82·89	83·09	83·48	83·78	84·08	84·39	84·78	85·08	85·39	85·77
79·0	80·33	80·72	81·01	81·22	81·62	81·91	82·10	82·51	82·81	83·09	83·29	83·68	83·98	84·29	84·59	84·98	85·29	85·59	85·97
79·2	80·53	80·92	81·22	81·42	81·82	82·11	82·31	82·71	83·01	83·30	83·50	83·88	84·18	84·49	84·79	85·18	85·49	85·79	86·17
79·4	80·73	81·12	81·42	81·62	82·02	82·32	82·51	82·91	83·22	83·50	83·70	84·08	84·39	84·69	84·99	85·39	85·69	85·99	86·38
79·6	80·93	81·32	81·62	81·82	82·23	82·52	82·71	83·11	83·42	83·70	83·90	84·28	84·59	84·89	85·19	85·59	85·89	86·20	86·58
79·8	81·13	81·52	81·82	82·02	82·43	82·72	82·91	83·32	83·62	83·90	84·10	84·49	84·79	85·09	85·40	85·79	86·09	86·40	86·78
80·0	81·34	81·73	82·02	82·23	82·63	82·92	83·11	83·52	83·82	84·10	84·30	84·69	84·99	85·30	85·60	85·99	86·30	86·60	86·98
80·2	81·54	81·93	82·23	82·43	82·83	83·12	83·32	83·72	84·02	84·31	84·51	84·89	85·19	85·50	85·80	86·19	86·50	86·80	87·18
80·4	81·74	82·14	82·43	82·63	83·03	83·33	83·52	83·92	84·23	84·51	84·71	85·09	85·40	85·70	86·00	86·40	86·70	87·00	87·39
80·6	81·94	82·34	82·63	82·83	83·24	83·53	83·72	84·12	84·43	84·71	84·91	85·20	85·60	85·90	86·21	86·60	86·90	87·21	87·59
80·8	82·14	82·54	82·83	83·03	83·44	83·73	83·92	84·33	84·63	84·91	85·11	85·30	85·80	86·10	86·41	86·80	87·10	87·41	87·79
81·0	82·35	82·74	83·03	83·24	83·64	83·93	84·12	84·53	84·83	85·11	85·31	85·50	86·00	86·31	86·61	87·00	87·31	87·61	87·99
81·2	82·55	82·94	83·24	83·44	83·84	84·13	84·33	84·73	85·03	85·32	85·52	85·70	86·21	86·51	86·81	87·21	87·51	87·81	88·19
81·4	82·75	83·14	83·44	83·64	84·04	84·34	84·53	84·93	85·24	85·52	85·72	85·90	86·41	86·71	87·01	87·41	87·71	88·01	88·40
81·6	82·95	83·35	83·64	83·84	84·25	84·54	84·73	85·13	85·44	85·72	85·92	86·10	86·61	86·91	87·22	87·61	87·91	88·22	88·60
81·8	83·15	83·55	83·84	84·04	84·45	84·74	84·93	85·34	85·64	85·92	86·12	86·31	86·81	87·11	87·42	87·81	88·11	88·42	88·80
82·0	83·36	83·75	84·04	84·25	84·65	84·94	85·13	85·54	85·84	86·02	86·33	86·51	87·01	87·32	87·62	88·01	88·32	88·62	89·00
82·2	83·56	83·95	84·25	84·45	84·85	85·14	85·34	85·74	86·04	86·23	86·53	86·71	87·22	87·52	87·82	88·22	88·52	88·82	89·21
82·4	83·76	84·16	84·45	84·65	85·05	85·35	85·54	85·94	86·25	86·43	86·73	86·91	87·42	87·72	88·02	88·42	88·72	89·02	89·41
82·6	83·96	84·36	84·65	84·85	85·26	85·55	85·74	86·14	86·45	86·63	86·93	87·11	87·62	87·92	88·23	88·62	88·92	89·23	89·61
82·8	84·16	84·56	84·85	85·05	85·46	85·75	85·94	86·35	86·65	86·83	87·13	87·31	87·82	88·12	88·43	88·82	89·12	89·43	89·81
83·0	84·37	84·76	85·05	85·26	85·66	85·95	86·14	86·55	86·85	87·03	87·34	87·51	88·02	88·33	88·63	89·02	89·32	89·63	90·01
83·2	84·57	84·96	85·26	85·46	85·86	86·15	86·35	86·75	87·05	87·24	87·54	87·71	88·23	88·53	88·83	89·23	89·52	89·83	90·22
83·4	84·77	85·16	85·46	85·66	86·06	86·36	86·55	86·95	87·26	87·44	87·74	87·91	88·43	88·73	89·03	89·43	89·72	90·03	90·42
83·6	84·97	85·37	85·66	85·86	86·27	86·56	86·75	87·15	87·46	87·64	87·94	88·12	88·63	88·93	89·24	89·63	89·92	90·24	90·62
83·8	85·17	85·57	85·86	86·06	86·47	86·76	86·95	87·36	87·66	87·84	88·14	88·32	88·83	89·13	89·44	89·83	90·12	90·44	90·82
84·0	85·38	85·77	86·06	86·27	86·67	86·96	871·5	87·56	87·86	88·04	88·35	88·52	89·03	89·34	89·64	90·03	90·33	90·64	91·02

(32A) BAROMETRIC PRESSURE CORRECTION

740.5 Mm	742 Mm	743 Mm	744.5 Mm	745.5 Mm	747 Mm	748 Mm	749.5 Mm	751 Mm	752 Mm	753 Mm	754.5 Mm	756 Mm	757 Mm	758 Mm	760 Mm
81·93	81·78	81·63	81·49	81·35	81·22	81·08	80·94	80·80	80·67	80·53	80·39	80·26	80·12	79·99	79·80
82·21	82·06	81·91	81·77	81·63	81·50	81·36	81·22	81·08	80·95	80·81	80·67	80·54	80·40	80·27	80·08
82·48	82·33	82·18	82·04	81·90	81·77	81·63	81·49	81·35	81·22	81·08	80·94	80·81	80·67	80·53	80·35
82·76	82·61	82·46	82·32	82·18	82·05	81·91	81·77	81·63	81·50	81·36	81·22	81·09	80·95	80·82	80·63
83·04	82·89	82·74	82·60	82·46	82·33	82·19	82·05	81·91	81·78	81·64	81·50	81·37	81·23	81·10	80·91
83·32	83·17	83·02	82·88	82·74	82·61	82·47	82·33	82·19	82·06	81·92	81·78	81·65	81·51	81·38	81·19
83·60	83·45	83·30	83·16	83·02	82·89	82·75	82·61	82·47	82·34	82·20	82·06	81·93	81·79	81·66	81·47
83·88	83·73	83·58	83·44	83·30	83·17	83·03	82·89	82·75	82·62	82·48	82·34	82·21	82·07	81·94	81·74
84·15	84·00	83·85	83·77	83·57	83·44	83·30	83·16	83·02	82·89	82·75	82·62	82·48	82·34	82·21	82·02
84·43	84·28	84·13	83·99	83·85	83·72	83·58	83·44	83·30	83·17	83·03	82·89	82·76	82·62	82·49	82·30
84·72	84·57	84·42	84·28	84·14	84·01	83·87	83·73	83·59	83·46	83·32	83·18	83·05	82·91	82·78	82·59
85·00	84·85	84·70	84·56	84·42	84·29	84·15	84·01	83·87	83·74	83·60	83·46	83·33	83·19	83·06	82·87
85·28	85·13	84·98	84·84	84·70	84·57	84·43	84·29	84·15	84·02	83·88	83·74	83·61	83·47	83·31	83·15
85·56	85·41	85·26	85·12	84·98	84·85	84·71	84·57	84·43	84·30	84·16	84·02	83·89	83·75	83·62	83·43
85·83	85·68	85·53	85·39	85·25	85·12	84·98	84·84	84·70	84·57	84·43	84·29	84·16	84·02	83·89	83·70
86·12	85·97	85·82	85·76	85·53	85·40	85·26	85·12	84·98	84·85	84·71	84·57	84·41	84·30	84·17	83·98
86·40	86·25	86·10	86·01	85·81	85·68	85·54	85·40	85·26	85·13	84·99	84·85	84·72	84·58	84·45	84·26
86·68	86·53	86·38	86·32	86·09	85·96	85·82	85·68	85·54	85·41	85·27	85·13	85·00	84·86	84·73	84·54
86·96	86·81	86·66	86·60	86·37	86·24	86·10	85·96	85·82	85·69	85·55	85·41	85·28	85·14	85·01	84·82
87·24	87·09	86·94	86·88	86·65	86·52	86·38	86·24	86·10	85·97	85·83	85·79	85·56	85·42	85·29	85·10
87·52	87·37	87·22	87·16	86·93	86·80	86·66	86·52	86·38	86·25	86·11	85·97	85·84	85·70	85·57	85·38
87·80	87·65	87·50	87·44	87·21	87·08	86·94	86·80	86·66	86·53	86·39	86·25	86·12	85·98	85·85	85·66
88·08	87·93	8 ·78	87·72	87·49	87·36	87·22	87·08	86·94	86·81	86·67	86·53	86·40	86·26	86·13	85·94

BAROMETRIC PRESSURE CORRECTION.

$CaCO_3$ per cent	762 Mm	763 Mm	764.5 Mm	766 Mm	767 Mm	768 Mm	769.7 Mm	771 Mm	772.2 Mm	773.5 Mm	774.7 Mm	776 Mm	777.2 Mm	778.5 Mm	779.7 Mm	781 Mm
71·50	79·67	79·53	79·39	79·26	79·12	78·99	78·80	78·67	78·53	78·39	78·26	78·12	77·99	77·80	77·61	77·53
71·75	79·95	79·81	79·67	79·54	79·40	79·27	79·08	78·95	78·81	78·67	78·54	78·40	78·27	78·08	77·95	77·71
72.00	80·22	80·08	79·94	79·81	79·67	79·53	79·35	79·22	79·08	78·94	78·81	78·67	78·54	78·35	78·22	78·08
72·25	80·50	80·36	80·22	80·09	79·95	79·82	79·63	79·50	79·36	79·22	79·09	78·95	78·82	78·63	78·50	78·36
72·50	80·78	80·64	80·50	80·37	80·23	80·10	79·91	79·78	79·64	79·50	79·37	79·23	79·10	78·91	78·78	78·64
72·75	81·06	80·92	80·78	80·65	80·51	80·38	80·19	80·06	79·92	79·78	79·65	79·52	79·38	79·19	79·06	78·92
73·00	81·34	81·20	81·06	80·93	80·79	80·66	80·47	80·34	80·20	80·06	79·93	79·79	79·66	79·47	79·34	79·20
73·25	81·62	81·48	81·34	81·21	81·07	80·94	80·75	80·62	80·48	80·34	80·21	80·07	79·94	79·75	79·62	79·48
73·50	81·89	81·75	81·61	81·48	81·34	81·21	81·02	80·89	80·75	80·61	80·48	80·34	80·21	80·02	79·89	79·75
73·75	82·17	82·03	81·89	81·76	81·62	81·49	81·30	81·17	81·03	80·89	80·76	80·62	80·49	80·30	80·17	80·03
74·00	82·46	82·32	82·18	82·05	81·91	81·78	81·59	81·46	81·32	81·18	81·05	80·91	80·78	80·59	80·46	80·32
74·25	82·74	82·60	82·46	82·33	82·19	82·06	81·87	81·74	81·60	81·46	81·33	81·19	81·06	80·87	80·74	80·60
74·50	83·02	82·88	82·74	82·61	82·47	82·34	82·15	82·02	81·88	81·74	81·61	81·47	81·34	81·15	81·02	80·88
74·75	83·36	83·16	83·02	82·89	82·75	82·62	82·43	82·38	82·16	82·02	81·99	81·75	81·62	81·43	81·30	81·16
75·00	83·57	83·43	83·29	83·16	83·02	82·89	82·70	82·57	82·43	82·29	82·16	82·02	81·89	81·70	81·57	81·43
75·25	83·85	83·71	83·57	83·44	83·30	83·17	82·98	82·85	82·71	82·57	82·44	82·30	82·17	81·98	81·85	81·71
75·50	84·13	83·99	83·85	83·72	83·58	83·45	83·26	83·13	82·99	82·85	82·72	82·58	82·45	82·26	82·13	81·99
75·75	84·41	84·27	84·13	84·00	83·86	83·73	83·54	83·41	83·27	83·13	83·00	82·86	82·73	82·54	82·41	82·27
76·00	84·69	84·55	84·41	84·28	84·14	84·01	83·82	83·69	83·55	83·41	83·28	83·14	83·01	82·82	82·69	82·55
76·25	84·97	84·83	84·69	84·56	84·42	84·29	84·10	83·97	83·83	83·69	83·56	83·42	83·29	83·10	82·97	82·83
76·50	85·25	85·11	84·97	84·84	84·70	84·57	84·38	84·25	84·11	83·97	83·84	83·70	83·57	83·38	83·25	83·11
76·75	85·53	85·39	85·25	85·12	84·98	84·85	84·66	84·53	84·49	84·25	84·12	83·98	83·85	83·66	83·53	83·39
77·00	85·81	85·67	85·53	85·40	85·26	85·13	84·94	84·81	84·77	84·53	84·40	84·26	84·13	83·94	83·81	83·67

(33A) TYPICAL ANALYSES.

CHALK (Dried at 105°)		LIME-STONE (Oolitic)	LIME-STONE (Siliceous)	LIME-STONE (Magnesian)	HYDRAULIC LIME-STONE	NATURAL CEMENT STONE
Silica (SiO_2)	1·51	1·64	8·02	6·62	22·82	13·56
Alumina (Al_2O_3) Ferric oxide (Fe_2O_3)	0·42	1·21	2·62	1·81	9·76	8·00
Lime (CaO)	54·62	54·56	48·46	30·44	33·71	42·05
Magnesia (MgO)	0·13	0·35	1·00	17·48	1·27	0·54
Loss on ignition (CO_2H_2O)	42·90	42·23	38·86	42·92	31·58	34·34
Alkalies and loss (by diff.)	·42	—	1·01	0·70	0·86	1·51
	100·00	100·00	100·00	100·00	100·00	100·00

CLAYS, ETC., SUITABLE FOR CEMENT.

RIVER MUD (Dried at 105°)

Silica (SiO_2)	26·85	SiO_2	= 2·47
Insoluble (sand)	30·29	Al_2O_3	= 0·70
		K_2O	= 0·63
Alumina (Al_2O_3)	16·68	Quartz	= 26·49
Ferric oxide (Fe_2O_3)	6·60		
Lime (CaO)	1·96		
Magnesia (MgO)	2·14		
Loss on ignition	11·94		
Alkalies and loss (by diff.)	3·54		
	100·00		

CLAY (Holland)

Silica (SiO_2)	26·86	SiO_2	= 7·52
Insoluble (sand)	39·75	Al_2O_3	= 2·15
		K_2O	= 1·93
Alumina (Al_2O_3)	13·96	Sand	= 28·15
Ferric oxide (Fe_2O_3)	6·58		
Lime (CaO)	1·55		
Magnesia (MgO)	1·59		
Sulphuric anhydride (SO_3)	0·27		
Sulphur as Sulphide	0·06		
Loss on ignition	7·99		
	98·61		

CLAY (Ultimate Analysis)

Silica (SiO_2)	67·14
Alumina (Al_2O_3)	14·96
Ferric oxide (Fe_2O_3)	6·50
Lime (CaO)	2·45
Magnesia (MgO)	1·92
Loss on ignition	5·44
Alkalies and loss	1·59
	100·00

SHALE

Silica (SiO_2)	63·45
Alumina (Al_2O_3)	17·25
Ferric oxide (Fe_2O_3)	4·25
Lime (CaO)	nil
Magnesia (MgO)	1·93
Loss on ignition	10·25
Alkalies and loss	2·87
	100·00

CALCAREOUS CLAY

Silica (SiO_2)	9·08	SiO_2	= 0·63
Insoluble (sand)	20·45	Al_2O_3	= 0·18
		K_2O	= 0·16
Alumina (Al_2O_3)	6·48	Quartz	= 19·48
Ferric oxide (Fe_2O_3)	2·40		
Lime (CaO)	32·32		
Magnesia (MgO)	1·12		
Loss on ignition	26·24		
Alkalies and loss (by diff.)	1·91		
	100·00		

PUZZOLANIC MATERIAL.

SiO_2	44·91
Al_2O_3	17·92
Fe_2O_3	8·40
MnO	0·83
CaO	10·48
MgO	4·81
Loss on ignition	7·76
Sulphur	traces
Alkalies and loss (by diff.)	1·89
	100·00

ANALYSES OF TYPICAL SLURRIES AND RAW MEALS.

	DRY SLURRY, CHALK, AND MUD.	LIMESTONE AND CLAY.	LIMESTONE AND SHALE.
Silica and insoluble	14·33	14·40	14·40
Ferric oxide and alumina	6·20	6·36	5·72
Lime (CaO)	42·34	42·01	42·40
Magnesia (MgO)	0·48	0·52	1·20
Loss on ignition	34·68	34·92	34·98
Alkalies and loss	1·97	1·79	1·30
	100·00	100·00	100·00

ANALYSES OF TYPICAL CEMENTS.

	FROM BOTTLE KILNS.	CHAMBER KILNS.	ROTARY.	FROM LIMESTONE AND ROCK	FROM LIMESTONE AND SHALE	FROM LIMESTONE AND SHALE.	FROM SILICEOUS LIMESTONE AND CLAY-STONE RICH IN IRON OXIDE.
Silica (SiO_2)	22·64	22·66	22·88	24·46	19·04	24·54	20·94
Insoluble	1·16	1·22	0·36	0·56	0·24	0·54	1·26
Alumina (Al_2O_3)	13·68	10·92	5·18	10·41	12·12	10·90	7·22
Ferric oxide (Fe_2O_3)			3·62				4·98
Lime (CaO)	57·90	60·43	63·51	60·38	62·94	60·29	61·35
Magnesia (MgO)	1·12	1·07	1·33	1·12	1·38	2·18	2·14
Sulphuric anhydride (SO_3)	1·97	1·72	0·91	0·65	1·13	nil	0·83
Loss on ignition	0·32	0·92	1·36	0·72	2·18	0·38	0·70
Alkalies and loss (by diff.)	1·21	1·06	0·85	1·67	0·97	1·17	0·58
	100·00	100·00	100·00	100·00	100·00	100·00	100·00

RESULT OF ANALYSES OF TYPICAL WATERS, IN GRAINS PER GALLON.

Chlorine as chlorides .	1·05	2·1	5·6	2·45	1·00	5·95
"Free" ammonia . .	Minute trace	nil	trace	nil	·001	·056
"Albuminoid" amm.	nil	·0028	·0014	·0014	·004	·0084
Total solids	27·2	27·0	54·87	21·6	3·5	100
Colour of ditto . . .	White	slight yellow tinge	brown, hygroscopic	light yellow	faintly yellow	white
Nitric acid as nitrates	nil	nil	7·7	1·6	nil	nil
Equal to nitrogen . .	nil	nil	1·7	·35	nil	nil
Oxygen required to oxidise decomposing matter . . .	·0056	nil	·0084	·0028	·002	nil
Sediment and microscopic results . .	Little vegetable deposit	No deposit	gelatinous fungus.	Slight sediment. Decaying vegetable matter. Mycelium of a fungus. Rotifers and streptococci.	nil	nil
Colour of water . .	colourless	—	—	—	faint blue	clear bluish
Temporary hardness .	—	15°	20°	—	2·5	34°
Permanent " .	—	5°	5°	—		
Report :—	An excellent drinking water.	Good water with little surface drainage	Highly polluted with sewage. Unfit for use.	Polluted with rain water from a dirty roof	Excellent, but should not be kept in leaden cistern.	A pure water, but too highly mineralised for domestic use.
Character of source :—	From well over 30ft. deep	As preceding, but well unused for some time.	Pump near kitchen door.	Good water from deep well.	Silurian strata.	From 160ft. bore through "Forest Marble"

(34A)

TABLE FOR USE WITH BLOUNT'S FLASK.

SPECIFIC GRAVITY. USING 50 GRAMS OF CEMENT AND 50 c.c. OF OIL.

Volume of oil displaced.	Specific Gravity.	Volume of Oil displaced.	Specific Gravity.
15·00	3·333	15·90	3·145
15·10	3·312	15·95	3·135
15·20	3·290	16·00	3·125
15·30	3·268	16·05	3·115
15·40	3·246	16·10	3·105
15·50	3·225	16·15	3·095
15·55	3·215	16·20	3·086
15·60	3·205	16·25	3·077
15·65	3·195	16·30	3·067
15·70	3·185	16·35	3·058
15·75	3·175	16·40	3·049
15·80	3·165	16·45	3·039
15·85	3·155	16·50	3·030

$$\text{Sp. gr.} = \frac{50}{\text{c.c. displaced}}$$

(35A) Table for Use with Anderson's Specific Gravity Bottle using 150 grams of Cement and 200 c.c. of Oil.

Displacement.	Specific gravity.	Displacement.	Specific gravity.	Displacement	Specific gravity.
45·0	3·333	49·0	3·061	53·0	2·830
·1	3·326	·1	3·055	·1	2·824
·2	3·319	·2	3·049	·2	2·820
·3	3·311	·3	3·043	·3	2·814
·4	3·304	4	3·036	·4	2·809
·5	3·297	·5	3·030	·5	2·804
·6	3·289	·6	3·024	·6	2·798
·7	3·282	·7	3·018	·7	2·793
·8	3·275	·8	3·012	·8	2·788
·9	3·268	·9	3·006	·9	2 782
46·0	3·261	50 0	3·000	54·0	2·777
·1	3·254	·1	2·994	·1	2·772
·2	3·247	·2	2·988	·2	2 767
·3	3 240	·3	2·982	·3	2·762
·4	3·233	·4	2·976	·4	2·757
5	3·226	·5	2·970	·5	2·752
·6	3·219	6	2·964	·6	2·747
·7	3·212	·7	2·959	·7	2·742
·8	3·205	·8	2·953	·8	2·737
·9	3·198	·9	2·947	·9	2·732
47·0	3·191	51·0	2·941	55·0	2·727
·1	3·185	·1	2·935	·1	2·722
2	3·178	·2	2·930	·2	2·718
·3	3·171	·3	2·924	·3	2·712
·4	3·165	·4	2·918	·4	2·707
·5	3·158	·5	2·913	·5	2·702
·6	3·151	·6	2·907	·6	2·697
·7	3·145	·7	2·901	·7	2·693
·8	3·138	·8	2·896	·8	2·688
·9	3·132	·9	2·890	·9	2·683
48·0	3·125	52·0	2·885	56·0	2·678
·1	3·119	·1	2·879	·1	2·673
·2	3·112	·2	2 874	·2	2·669
·3	3·106	·3	2·868	·3	2 664
·4	3·099	·4	2·863	·4	2·659
·5	3·093	·5	2·857	·5	2·654
·6	3·086	·6	2 852	·6	2·650
·7	3·080	·7	2·846	·7	2·645
·8	3·074	·8	2·841	·8	2·640
·9	3·068	·9	2·836	·9	2·636

$$\text{Sp. gr.} = \frac{150}{\text{c.c. displaced}}$$

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